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TECHNICAL REPORT 7714

CRITERIA FOR THE PRODUCTION OF HIGH PURITY WATER

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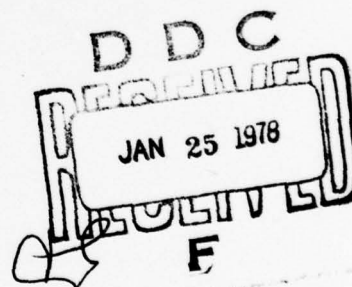
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Information is presented to supplement formal criteria needed for designing or choosing high purity water systems. Type of information presented includes a discussion of water quality and summaries of commonly used water quality criteria; description and design considerations about ion exchange, reverse osmosis, distillation, filtration, and carbon adsorption operations; description and design considerations about distribution and storage of high purity water to include materials of construction and centralized versus

20. Abstract

decentralized systems. Other information includes discussion of high purity water uses in medical facilities and laboratories and control of high purity water quality. A bibliography is included in the report.

ACKNOWLEDGMENT

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INTRODUCTION AND APPROACH

Design of high purity water systems is successful only when the quality and quantity of the high purity water desired are defined and when factors which may influence design are recognized and understood. It is to this end that the information in this report is compiled.

The term "high purity water" used throughout the report is defined as any water of a quality superior to that defined in the National Interim Primary Drinking Water Regulations, or any potable water receiving additional treatment (such as water softening). A summary of the Regulations is in Table 1 with appropriate reference. "High purity water system" is defined to consist of high purity water production, distribution, and storage facilities.

This report is not a design manual. Specific design details are not addressed. The information presented is intended to supplement technical and other criteria used for design. This report does define or identify items which should be considered in design. To that extent it will overlap to some degree with formal design criteria.

There is no best high purity water treatment method for the general situation. Final selection of a treatment system will be a function of particular circumstances. An effort is made in this report to outline the options which are available in order to give the designer a full array of viable alternatives. Considering all the factors, being aware of the options, and deciding upon the quantity and quality of high purity to be produced will result in the best design for the production of high purity water.

OBJECTIVE

The objective of this report is to present information which should be considered during the design or specification of high purity water production systems in health care facilities and laboratories.

WATER QUALITY

General

The ultimate objective of producing a known quality high purity water is to insure the health of the user is not threatened and/or that the accuracy of analytical tests is not impaired. A second objective related to the first is to supply acceptable quality water to user equipment to allow it to perform its primary function satisfactorily and efficiently. An example of direct-user equipment is the kidney dialysis machine. An

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TABLE 1.   SUMMARY OF NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

A.   MAXIMUM CONTAMINANT LEVELS (MCL) - INORGANIC CHEMICALS

Chemical	MCL (mg/l)
Arsenic	0.05
Barium	1.0
Cadmium	0.010
Chromium	0.05
Lead	0.05
Mercury	0.002
Nitrate (as N)	10.0
Selenium	0.01
Silver	0.05

Fluoride (naturally occurring)

53.7°F and below	2.4
53.8 - 58.3°F	2.2
58.4 - 63.8°F	2.0
63.9 - 70.6°F	1.8
70.7 - 79.2°F	1.6
79.3 - 90.5°F	1.4

B.   MAXIMUM CONTAMINANT LEVELS (MCL) - ORGANIC CHEMICALS

Chemical	MCL (mg/l)
Chlorinated hydrocarbons	0.0002
Endrin	0.004
Lindane	0.1
Methoxychlor	0.1
Toxaphene	0.005

Chlorophenoxys

2,4-D	0.1
2,4,5-TP (Silvex)	0.01

C.   MAXIMUM CONTAMINANT LEVELS (MCL) - RADIONUCLIDES

Chemical	MCL (picocuries/l)
Natural	
Gross alpha activity	15
Radium 226 + radium 228	5

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Chemical	MCL (picocuries/1)
Man-made	
Gross beta activity	50
Tritium	20,000
Strontium-90	8

D. MAXIMUM CONTAMINANT LEVELS - TURBIDITY

Monthly average - 1 turbidity unit (TU) or 5 TUs with state approval provided it does not interfere with disinfection, maintenance of chlorine residual, or bacteriological testing.

2-Day average - 5 TUs.

E. MAXIMUM CONTAMINANT LEVELS - MICROBIOLOGICAL CONTAMINANTS[a]

Coliform Method	Per Month	Less than 20 Samples per Month	More than 20 Samples per Month
Membrane filter	Number of coliform bacteria shall not exceed:		
(100 ml portions)	1/100 ml Average density	4/100 ml in one sample	4/100 ml in 5% of all samples
Multiple tube fermentation	Coliform bacteria shall not be present in more than:		
	10% of portions	3 portions in one sample	3 portions in 5% of samples

a. The regulation allows substitution of chlorine residual readings for bacteriological tests when state approved. See regulation for details.

NOTE: Consult the regulation for detailed sampling frequencies, reporting, and record keeping requirements.

Regulation: Environmental Protection Agency - Water Programs "National Interim Primary Drinking Water Regulations," Federal Register, Part IV, Vol. 40, No. 248, pp. 59566-59588 (December 24, 1975) and Environmental Protection Agency - Drinking Water Regulations "Radionuclides," Federal Register, Part II, Vol. 41, No. 133, pp. 28402-28405 (July 9, 1976).

example of indirect-user equipment is an atomic adsorption spectro-photometer. A less obvious example of indirect-user equipment is an ion exchange column or distillation unit.

When designing high purity water production systems, their water pretreatment requirements are often overlooked. As a result, such indirect-user equipment (including storage and transfer facilities) perform unsatisfactorily and the primary objective of health or analytical accuracy is compromised. The poor performance of this equipment is normally due to failure of removing impurities which impede its operation. These impurities cause unwanted corrosion, deposition, structural defects, and ultimately malfunctions. They cause erroneous measurements by analytical equipment.

The section discussing unit operations identifies common impurities which affect indirect user equipment (high purity water treatment equipment. The section discussing uses of water for medical facilities and laboratories does the same for direct user equipment. The section discussing storage and distribution takes the reverse approach. It discusses how the indirect user equipment (distribution lines and storage tanks) affect the purity of the water. The reader is referred to these sections for specifics. It is important to remember that water - especially high purity water - is an excellent solvent. It has a tendency to dissolve components of structures containing it and the atmosphere surrounding it.

Influent Quality

Influent quality refers to quality of the source water entering the high purity water treatment train. The most apparent reason to characterize influent water quality is to allow proper selection and sizing of high purity water treatment equipment. When this is done the effluent high purity water meets specified criteria and may be used as intended.

An equally important reason to characterize influent water quality is to identify and quantify impurities or conditions which adversely affect operation of the treatment equipment itself. A summary of those factors which commonly affect high purity water operations is given in Table 2. This table is not intended to be all encompassing but rather highlights some important considerations.

The multiple considerations about high purity water use and proper high purity water production equipment design require a thorough analysis of the influent water. The influent water must be fully characterized to insure that all water production equipment design options are realized and the most efficient design is adopted. Table 3 lists common components of a desirable influent water quality analysis. Analyses of several samples taken over a period of time is necessary to insure that the water

TABLE 2.   IMPURITIES AFFECTING THE EFFICIENCY OF SEVERAL
WATER TREATMENT OPERATIONS[a]

Water Treatment Operation	Impurity or Condition
Reverse osmosis	High temperature
	High and low pH
	Suspended solids
	Bacteria
	Hardness
	Chlorine (for polyamide membranes)
Ion exchange	Suspended solids
	Bacteria
	Organics
	Iron
	Silica
	Carbon dioxide
Distillation	Hardness
	Alkalinity
	Carbon dioxide
Carbon adsorption	Suspended solids
	Bacteria
	High pH
Filtration	Suspended solids

a.   In sufficient quantities or at extreme values these impurities may
coat, deposit, foul, corrode, interfere with the primary function,
contaminate, or otherwise affect the treatment operation efficiency.

characterized is representative of that to be expected at the site. If a
surface source of water is used, characterization spanning each season of
the year is desirable. Detailed analyses of non-representative water
samples are of no value. When possible, comparison of levels of impuri-
ties measured with data available at the tap water treatment plant is
desirable to create a confidence that the water samples are indeed repre-
sentative. Although the types of data gathered at water treatment plants
are limited, the measurements taken are usually made routinely over long
periods of time which is the true test of water characterization.

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TABLE 3.   SUGGESTED INFLUENT WATER QUALITY ANALYSES[a]

Cations	Anions	Other[b]
Calcium	Bicarbonate	Methyl orange (total) alkalinity[c]
Magnesium	Carbonate	Phenolphthalein alkalinity[c]
Sodium	Hydroxyl	pH range
Manganese	Sulfate	Turbidity
Potassium	Chloride	Total dissolved solids
Iron	Nitrate	Hardness
	Silica (as SiO_2)	Suspended solids
	Free carbon dioxide	Chlorine residual
	Phosphate	Temperature range
	Fluoride	Color
Total cations (as $CaCO_3$)		Total organic carbon
		Standard plate count

a. Methods for the analyses of these constituents may be found in "Standard Methods for the Examination of Water and Wastewater" (American Public Health Association) or "Methods for Chemical Analyses of Water and Wastes" (Environmental Protection Agency).
b. Other tests measuring sanitary quality, wastewater strength, and tests measuring heavy metals should also be considered.
c. Methyl orange and phenolphthalein alkalinity encompass bicarbonate, carbonate and hydroxyl anion concentration.

Effluent Quality

Effluent quality refers to quality of the water leaving the high purity water treatment train. The effluent is the high purity water. The purity level requirements of the water depend on the intended use or the selected quality criteria. For industrial activities and to a lesser extent analytical activities, the process or procedure using the high purity water establishes which impurities cannot be tolerated. For such cases, treatment design and effluent standards are centered on them. For multiple uses such as in health care or when there are a large number of analytical procedures and methods, broader, more general criteria are normally adopted (which in turn may require more elaborate and expensive treatment equipment to meet them). The most common water criteria are discussed below.

American Chemical Society (ACS) Standards. These Standards are designed to limit impurities most commonly interfering with chemical analysis and

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physical testing of water. Procedures for conducting all evaluation tests are described in the specifications. Methods of high purity water production are specified. ACS Standards are summarized in Table 4.

TABLE 4. SUMMARY OF AMERICAN CHEMICAL SOCIETY SPECIFICATIONS FOR REAGENT WATER (ACS STANDARDS)

A. WATER QUALITY REQUIREMENTS

Maximum specific conductance, micromho/cm at 25°C	2.0
Maximum silicate (as SiO_2), ppm	0.01
Maximum heavy metals (as Pb), ppm	0.01
Substances reducing permanganate	pass test

B. WATER PRODUCTION REQUIREMENTS

Water must be distilled or deionized.

Standard: "Reagent Chemicals-American Chemical Society Specifications," 5th ed., Washington, DC (1974).

American Society for Testing and Materials (ASTM) Standards. The ASTM Standards are also designed to limit impurities interfering with chemical and physical testing of water. They are more comprehensive than the ACS Standards in that they include a bacteriological quality requirement. Methods of test procedures are described. Detailed methods of high purity water preparation are specified. Uses for the various types of reagent grade water are discussed. ASTM Standards are summarized in Table 5.

College of American Pathologists (CAP) Specifications. CAP Water Specifications go beyond listing specifications and describing test procedures. The specifications also outline methods of quality control useful when testing, handling, and sampling the water. Frequency of testing is discussed, as is the significance of the several impurities listed. Recommended uses for the various types of reagent grade water are outlined in detail. The CAP Water Specifications are a useful, concise exercise in high purity water rational and criteria. They are especially appropriate for clinical and pathology laboratory work. Information contained in the specifications is valuable whether or not the specific impurity limiting criteria is followed. CAP Water Specifications are summarized in Table 6.

TABLE 5.   SUMMARY OF AMERICAN SOCIETY FOR TESTING AND MATERIALS
SPECIFICATIONS FOR REAGENT WATER (ASTM STANDARDS)

A. WATER QUALITY REQUIREMENTS

| Specification | Water Grade | | | |
	Type I	Type II	Type III	Type IV
Maximum total matter, mg/l	0.1	0.1	1.0	2.0
Maximum specific conductance micromho/cm at 25°C	0.06	1.0	1.0	5.0
Minimum specific resistance megohm-cm at 25°C	16.66	1.0	1.0	0.20
pH at 25°C	6.8-7.2	6.6-7.2	6.5-7.5	5.0-8.0
Minimum color retention time of potassium permanganate minutes	60	60	10	10
Bacteriological quality[a]	None specified	Sterile and pyrogen free as produced	None specified	None specified

B.   WATER TREATMENT, STORAGE AND DISTRIBUTION REQUIREMENTS

Preparation[b]

Pretreatment	Pretreat until distillation (treatment) <u>feed</u> has a max. conductance < 20 micromho/cm at 25°C.	As required	No	No
Treatment	Shall consist of distillation followed by polishing with a mixed bed of ion-exchange materials plus 0.20 micron membrane filters.	Distillation	Ion exchange or reverse osmosis or distillation followed by 0.45 micron membrane filters	Ion exchange osmosis or distillation or electro-dialysis

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Specification	Water Grade			
	Type I	Type II	Type III	Type IV
Storage	Block tin Quartz TFE-fluoro-carbon	Block tin Quartz TFE-fluorocarbon	Borosilicate glass polyethy-lene, PVC	Borosilicate glass, stainless steel polyethylene, PVC polyester-reinforced fiberglass
Protection from atmospheric gases	Yes	Yes	Yes	No

C. WATER USE RECOMMENDATIONS

	Used where maximum accuracy and precision is indicated	Used where a high degree of freedom from contami-nation is desired but where Type I water is not specified	Used for general laboratory testing	Used in pro-cedures requiring large amounts of water of moderate purity; particularly for the make up of synthetic test solutions

a. If sterility is required following storage, Type II water must be bottled and heated to 120°C for 20 minutes. Pyrogen-free Type II water must be tested in conformance with requirements of USP Standards. These precautions are taken to avoid possible contamination from storage.

b. All methods presume operation on a potable water supply free of suspended matter.

Standard: "Annual Book of ASTM Standards," Part 31, Water (Std. D-1193), American Society for Testing and Materials, pg. 40-42 (July 1974).

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TABLE 6. SUMMARY OF COLLEGE OF AMERICAN PATHOLOGISTS STANDARDS
FOR REAGENT GRADE WATER (CAP STANDARDS)

A. WATER QUALITY REQUIREMENTS

Specification[a]	Water Grade		
	Type I	Type II	Type III
Maximum specific conductance, micromho/cm	0.1	2	5
Minimum specific resistance, megohm-cm	10	0.5	0.2
Maximum silicate, mg/l	0.01	0.01	0.01
Maximum heavy metals, mg/l as Pb	0.01	0.01	0.01
Permanganate reduction	pass test	pass test	pass test

Specification	Level
Ammonia, mg/l	< 0.1
Carbon dioxide, mg/l[b]	< 3
Culture	Interpretation
Hardness[c]	Negative
pH	6.0 - 7.0
Sodium, mg/l	< 0.1 mg/l

B. WATER USE RECOMMENDATIONS

Type I	Type II	Type III
Atomic absorption, photometry, flame photometry, enzymology, electrolyte determination, inorganic ion determinations blood, gas, and pH determination, reference buffer solutions In general: for maximum accuracy and precision	May use where chemical procedures do not specifically require Type I water. May include many of procedures under Type I depending on circumstances except atomic adsorption. Most hematological, serological and microbiological procedures	General laboratory testing Most qualitative analysis procedures Most urinalysis, parasitology and histological procedures Rinsing glassware

a. Testing must be accomplished frequently enough to insure a constant supply of reagent grade water that meets the requirements and specifications.

b. Reagent water used in certain procedures should be free of carbon dioxide or dissolved gases.

c. CAP outlines a colorimetric screening test showing positive or negative results for the presence of Ca or Mg ions.

NOTE: CAP spells out additional details with respect to sample gathering, measurment, handling, storage, and frequency of testing.

Standard: A.R. Stier, L.K. Miller, and R.J. Smith, "Reagent Water," College of American Pathologists (CAP-2500-4-74), 3rd Printing, Chicago, IL (1974).

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United States Pharmacopeia (USP) Standards. Unlike other water stand-
ards, USP emphasizes pass/no pass or endpoint type tests rather than allow-
able concentrations (with a few exceptions). Furthermore, water is not
classified by type. It is classified by use. References to the USP occur
in several federal statutes, the most significant being the recognition of
the USP definitions and standards in the Food, Drug, and Cosmetic Act.
All tests are described by USP or an appropriate reference is given. The
USP water use classifications are described in detail and general treat-
ment operations are specified. USP Standards are summarized in Table 7.

National Committee for Clinical Laboratory (NCCLS) Standards. NCCLS
Standards represent what NCCLS believes to be the minimum quality for
water used in the clinical laboratory. They incorporate what are con-
sidered the best features of the ASTM, CAP, USP, and ASTM Standards. In
addition to outlining specifications and describing test procedures, NCCLS
Standards discuss frequency of testing, storage and handling procedures,
and define the scope of the impurities. The format is similar to that
used in the CAP Water Specifications. NCCLS Standards outline methods
for high purity water production and recommended uses for two types of
reagent grade water are discussed. NCCLS Standards are summarized in
Table 8.

Standard Methods for the Examination of Water and Wastewater.
Standard Methods discusses high purity water requirements for the prepa-
ration of culture media used in bacteriological examination of water.
Only distilled or demineralized water which has been tested and found
free from traces of dissolved metals and bactericidal or inhibitory com-
pounds is specified for preparation of culture media and reagents.
Although specific limiting concentrations are left to the discretion of
the investigator, the discussion names compounds which frequently are the
cause of nutrient contamination, examines their source, and suggests pre-
cautionary measures to be taken.

The Clinical Laboratories Improvement Act of 1967 does not have
requirements regarding specifications for high purity water. The act
addresses the areas of personnel standards, proficiency testing, and
quality control. This latter area has to do with insuring that adequate
facilities, equipment, and proper maintenance are available in the
laboratory.

Because these aforementioned criteria are designed to provide general
guidelines for a host of high purity water uses, they must be reviewed to
ascertain whether they adequately encompass specific situations. For
instance, specifications for bacteriological quality in the most common
high purity water quality criteria are limited or poorly defined. If
bacteriological quality is of prime importance, more detailed guidelines
may be needed. De Roos and Straub have outlined bacteriological quality

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TABLE 7. SUMMARY OF UNITED STATES PHARMACOPEIA XIX (USP STANDARDS)

A. WATER USE AND REQUIRED TESTS[a]

Water for Injection[b]	Bacteriostatic Water for Injection[c]	Sterile Water for Injection[d]	Sterile Water for Irrigation	Purified Water
Pyrogen	pH Chloride Total solids Antimicrobial agents Pyrogen Sterility	Chloride Oxidizable substances Total solids Pyrogen Sterility	Chloride Oxidizable substances Total solids Pyrogen Sterility	pH Chloride Sulfate Ammonia Calcium Carbon dioxide Heavy metals Oxidizable substances Total solids Bacteriological purity

B. WATER USE DEFINITIONS

Water for Injection - clear, colorless, odorless liquid. Intended for use as
    a solvent for the preparation of parenteral solution. Prepared by dis-
    tillation or reverse osmosis and contains no added substances.
    Category: Pharmaceutic aid (solvent).

Bacteriostatic Water for Injection - clear, colorless, odorless or having the
    odor of the antimicrobial substance. It is sterile water for injection
    containing one or more suitable antimicrobial agents. Prepared by distilla-
    tion or reverse osmosis. Category: Pharmaceutical aid (Sterile vehicle).

Sterile Water for Injection - clear, colorless, odorless liquid. It is water
    for injection sterilized and suitable packaged and contains no anti-
    microbial agent. Prepared by distillation or reverse osmosis.
    Category: Pharmaceutical aid (solvent).

Sterile Water for Irrigation - clear, colorless, odorless liquid. It is
    identical to sterile water for injection but has its peculiar packaging,
    storage, and labeling requirements. Category: Irrigating solution;
    pharmaceutical aid (solvent).

Purified Water - clear, colorless, odorless liquid. Cannot be used in prepara-
    tions intended for parenteral solutions. Prepared by distillation,
    ion-exchange, reverse osmosis or other suitable process. Category:
    Pharmaceutical aid (solvent).

a. All tests are described in USP or an appropriate reference is given. USP
    emphasizes pass/no pass or endpoint type tests rather than allowable
    concentrations.
b. In addition to those listed, it meets the requirements of the other tests
    under Purified Water with the exception of the test for bacteriological
    purity.
c. In addition to those listed, it meets the requirements of the other tests
    under Purified Water with the exception of those for ammonia, oxidizable
    substances, and bacteriological purity.
d. In addition to those listed, it meets the requirements of the other tests
    under Purified Water with the exception of the test for bacteriological
    purity.

Standard: "Pharmacopeia of the United States of America," 19th revision,
           United States Pharmacopeial Convention, Washington, DC (1975).

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TABLE 8. SUMMARY OF NATIONAL COMMITTEE FOR CLINICAL LABORATORY STANDARDS
(NCCLS STANDARDS)

A. WATER QUALITY REQUIREMENTS

Specification[a,b,c]	Type I	Type II
Total bacterial count, colonies/liter	10^4	10^5
Particulate matter, micrometers (maximum)	1.0	1.0
Permanganate time at 25°C, minutes (minimum)	60	10
pH	N/A	5.5 - 7.5
Resistivity at 25°C, megohms-cm	10	0.5
Silicate, mg/l SiO_2 (maximum)	0.05	0.1

B. WATER PRODUCTION REQUIREMENTS:

1. Type I water production may be based on any of the first three of the
following in combination with one of the last two processes:

a. distillation
b. deionization
c. reverse osmosis
d. activated carbon
e. membrane filtration

2. Type II water can be produced by any of the above methods provided it
meets specifications.

C. WATER USE RECOMMENDATIONS

1. Type I - Use in tests requiring minimum interference and maximum
accuracy such as:

a. Atomic adsorption spectrophotometry
b. Enzymatic procedures affected by silica or bacteria
c. Electrophoresis procedures
d. Buffer solutions (may also require CO_2 - free water)
e. Direct reading flame photometer procedures

2. Type II - Use for general laboratory testing and glassware rinsing.

a. All specifications are stated for water as measured at time of produc-
tion. The resistivity of Type I water must be measured in-line; all
other specifications relate to samples measured off-line.
b. The maximum time interval of the testing cycle for qualifying the
purity of the reagent water should be 1 week.
c. Manufacturers of in vitro diagnostic products and scientists using
clinical laboratory testing procedures should specify any additional
interferences which must be avoided such as heavy metals, ammonia,
and carbon dioxide.

Standard: "Reagent Water Specification and Test Methods for Water
Used in the Clinical Laboratory - Proposed Standard:
PSC-3," National Committee for Clinical Laboratory
Standards, Washington, DC (1976).

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criteria and uses for research and health care facilities.* Information
was based on an extensive review of the literature, interviews with scores
of investigators, and data collected on various types of high purity water
treatment systems. Table 9 outlines their suggested bacteriological cri-
teria for high purity water use.

Other water quality impurities which common high purity water criteria
generally ignore or are not specific about are heavy metal content, trace
organics, numbers and sizes of particles, and dissolved gases. If these
impurities can adversely affect intended high purity water use, additional
criteria need to be determined and suitable water production facilities
designed. Biomedical research laboratories must especially be aware of
such potential impurities when doing biological and biochemical research.
Such research includes growth of cell cultures, bacteria and fungi,
production of enzymes, analyses of amino acids, and microscopic studies
of tissues and cells.

UNIT OPERATIONS

General

This section contains information about the most common high purity
water treatment operations. Information about their performance, their
limitations, and other design and selection considerations are presented
so the potential user can better choose the treatment process best suited
for him. Within the discussions, the several unit operations are also
compared to each other.

In a given situation, two or three high purity production schemes or
treatment trains will appear to be able to produce the necessary high
purity water quality desired in the quantities needed. Traditional
engineering approaches then concentrate on the cost/benefit ratios of
each production scheme. Although dollar figures are not used, general
comparisons are made among the appropriate treatment operations with
respect to energy usage and the effect of influent water quality and
quantity on high purity water production.

The potential for expansion of or increased production by the treat-
ment operation and its associated cost must also be considered when
evaluating alternative treatment systems. Similarly, operation and
maintenance costs need to be taken into account in any specific situa-
tion. Operation and maintenance costs include considerations about
on-site control needed during treatment, costs of treatment, and
media replacement or regeneration. Degree of operator training, and

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* DeRoos, R.L. and C.P. Straub, "Study of Critical Quality Requirements
  for High Purity Water in Biomedical Research and Medical Care Facilities
  at the National Institutes of Health," NIH Research Report (Dec 1973).

TABLE 9. CATEGORIES OF BACTERIOLOGICAL QUALITY ON THE BASIS OF TOTAL PLATE COUNT

Water Grade	Total Numbers of Organisms/ml	Examples of Typical Water Treatment Systems	Examples of Possible Uses
1a	Sterile	Autoclaved, bottled	Inhalation therapy, irrigation
1b	Sterile/pyrogen free	Distillation (special still) followed by auto-claving	Injection, irrigation, some final rinses
2	1-10	Local still with pro-tected storage	Dialysate preparation
3	11-100	Building distilled water with heavy use	Most laboratory uses
4	101-1000	Building deionized water with heavy use	Most laboratory glassware final rinses
5	> 1000	Building deionized water poorly maintained	Some laboratory glassware washing

Source: De Roos, R.L. and C.P. Straub, "Study of Critical Quality Requirements for High-Purity Water in Biomedical Research and Medical Care Facilities at the National Institutes of Health," University of Minnesota Research Report, NIH Contract No. NIH-DRS-72-2111 (1973).

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safety and health aspects must also be taken into account. Information with respect to these considerations is discussed below.

Ion Exchange

Description

1) General. Ion exchange is the displacement of ions of one species attached to an insoluble exchange material by ions of a different species in solution when that solution is brought into contact with the exchange material. The exchange behaves as a chemically reversible interaction and exchange materials are regenerable.

The exchange material or ion exchange resin is made up of an elastic three dimensional hydrocarbon network (polymer) to which is attached a functional ion active group. The functional group is ionic and is balanced with an oppositely charged mobile anion or cation available for exchange.

The degree of acidity or basicity of a resin is controlled by using different functional groups. Similarly, the anion or cation available for exchange determines the type of regeneration.

Displacement of ions is a function of ionic concentration and chemical equilibria. For equal concentrations the relative order in which ions generally displace other ions is listed in Table 10.

2) Resin Classifications

a) Strongly Acidic Cation Exchangers. Such cation exchangers are characterized by their salt splitting ability. Sulfonic acid (HSO_3^-) is the functional group. The resin may be sodium or acid (hydrogen) regenerated and exchange works well in all pH ranges.

Sodium cycle ion exchange is used for water softening. Hardness producing ions of calcium and magnesium, and to a lesser extent ions of iron, manganese, and aluminum are exchanged for sodium ions on the resin. A typical reaction involving a resin (R^-) is:

$$CaSO_4 + 2Na^+R^- \leftrightarrow Ca^{++}R_2^- + Na_2SO_4$$
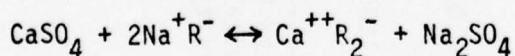
Ordinary salt is used for regeneration. Table 11 relates hardness to alkalinity as well as showing other associated relationships.

Hydrogen cycle ion exchange is used for water softening, dealkalizing, and demineralization. Cations in solution are exchanged for hydrogen ions on the resin, frequently with an increase in carbon dioxide. Typical reactions are:

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TABLE 10. DISPLACEMENT SERIES FOR AN
ION EXCHANGE MATERIAL[a]

Cations	Anions
La^{+++}	$SO_4^=$
Y^{+++}	$CrO_4^=$
Ba^{++}	NO_3^-
Sr^{++}	$A_SO_4^{\equiv}$
Ca^{++}	$PO_4^{\equiv}$
Mg^{++}	$MoO_4^=$
C_S^+	I^-
Rb^+	Cl^-
K^+	F^-
Na^+	OH^-
Li^+	
H^+	

a. For dilute solutions of equal concen-
    tration, ion species appearing low in
    the series are displaced from the
    exchange material by species listed
    high in the series.

TABLE 11. WATER HARDNESS RELATIONSHIPS

Cations   Anions

hardness[a] Ca^{++} carbonate or temporary hardness HCO_3^-
             Mg^{++} soft deposits $CO_3^=$ Alkalinity[b]

                                                                     OH^-

                    non-carbonate or permanent hardness
                    hard   hardness
                    Negative non-carbonate deposits
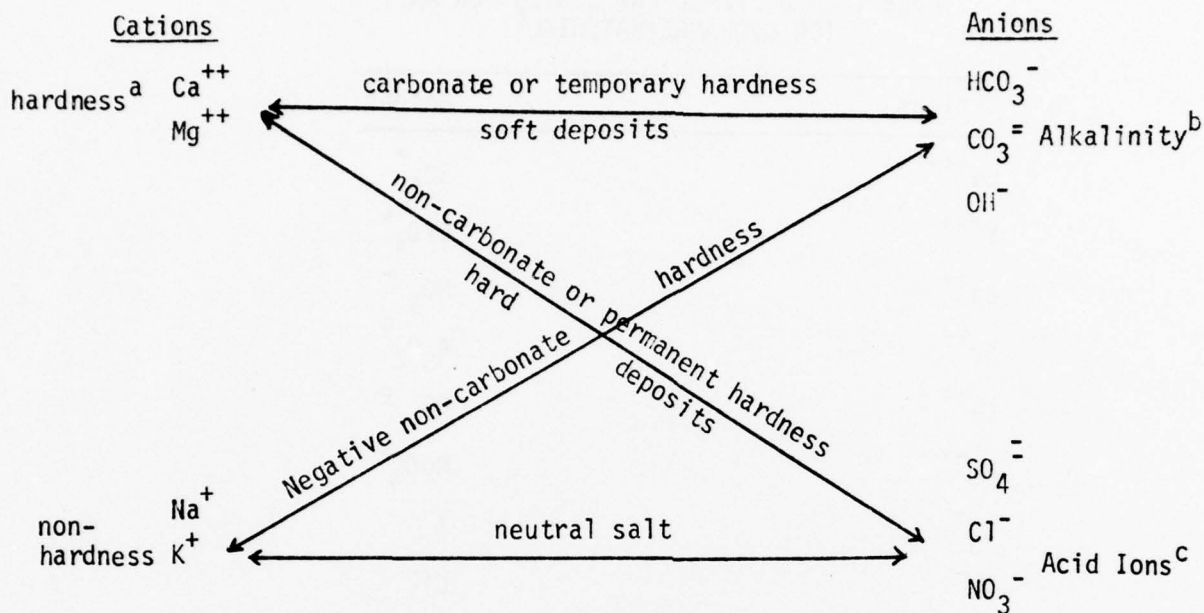
                                                                     $SO_4^=$
             Na^+ Cl^-
non- K^+ neutral salt   Acid Ions[c]
hardness NO_3^-

a. Also considered as minor hardness producing ions are iron and
    manganese.
b. Salts of weak acids and some biologically resistant organic acids
    (example, humic acid) also contribute to alkalinity.
c. These are hardness-associated anions which contribute to mineral
    acidity. Carbon dioxide is the principal source of natural
    water acidity.

Source: Fair, G.M. and J.C. Geyer, "Water Supply and Wastewater
         Disposal," John Wiley & Sons, Inc., New York (1967).

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$$Mg(HCO_3)_2 + 2H^+R^- \leftrightarrow Mg^{++}R_2^- + 2H_2CO_3 \leftrightarrow H_2O + CO_2$$

$$CaSO_4 + 2H^+R^- \leftrightarrow Ca^{++}R_2^- + H_2SO_4$$
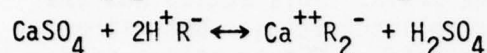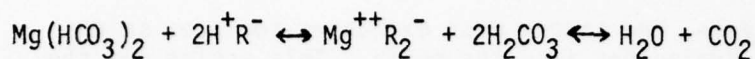
Effluent from the process has a depressed pH and will normally go through some process to raise pH such as anion exchange, neutralization, decarbonation, or deaeration. Carbon dioxide removal by vacuum deaeration is preferred over decarbonation because stripping increases the potential of introducing new impurities in the water and increases dissolved oxygen content (promoting corrosion). Regeneration is with a mineral acid and so safety precautions are needed to handle and store the acid. Sulfuric acid is not preferred because of precipitation problems which can occur as calcium sulfate, coating the resin. Operation of the process is sensitive to influent water quality but it does not add sodium to the water.
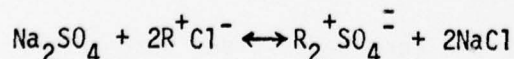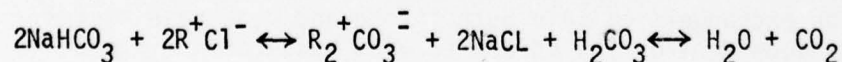
b) Weakly Acidic Cation Exchangers. Carboxylic groups (COOH) are normally the functional group. Weak acid cation exchangers are highly selective for multivalent cations over monovalent cations. They do not operate efficiently at pH less than five and, therefore, cannot split salts effectively. Weakly acidic cation exchangers are used primarily for softening and dealkalizing, usually in conjunction with a strongly acidic polishing resin. A typical reaction is:

$$Mg(HCO_3)_2 + 2H^+R^- \leftrightarrow Mg^{++}R_2^- + 2H_2CO_3 \leftrightarrow H_2O + CO_2$$
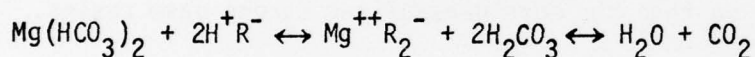
Treated effluent has a depressed pH and dissolved carbon dioxide. Depending on finished water criteria, pH may have to be neutralized or CO_2 may have to be removed as discussed previously. Regeneration is with a mineral acid and is more efficient than with strongly acidic exchangers.

c) Strong Base Anion Exchangers. Quaternary ammonium compounds serve as the functional group. The resin may be chloride or hydroxide regenerated.

In the chloride cycle, the strong base exchanger acts as a dealkalizer. Alkalinity of the water is removed without the use of an acid. Anions in solution are exchanged for chloride anions on the resin. Influent to this dealkalizing process usually needs to be softened because hard water gives marginal results. Typical reactions are:

$$2NaHCO_3 + 2R^+Cl^- \leftrightarrow R_2^+CO_3^= + 2NaCL + H_2CO_3 \leftrightarrow H_2O + CO_2$$

$$Na_2SO_4 + 2R^+Cl^- \leftrightarrow R_2^+SO_4^= + 2NaCl$$

Regeneration is with sodium chloride. Caustic soda (NaOH) may also be added to increase the pH which can become depressed from dissociation of carbonic acid. Significant concentrations of chloride ions in the feedwater lessen effectiveness of the resin by diminishing favorable equilibrium.

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In the hydroxide cycle, the strong base exchanger replaces anions in solution with hydroxide anions from the resin. This action has the effect of splitting neutral salts and neutralizing acids. Both strong and weak inorganic acids (such as carbonic acid as CO_2 or silica acid as SiO_2) may be removed if caustic soda is used for regeneration. Removal may proceed via ion exchange or absorption:

$$Na_2SO_4 + 2R^+OH^- \longleftrightarrow R_2^+SO_4^= + NaOH$$

$$H_2SO_4 + 2R^+OH^- \longleftrightarrow R_2^+SO_4^= + H_2O$$

$$CO_2 + R^+OH^- \longleftrightarrow R^+HCO_3^-$$
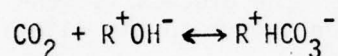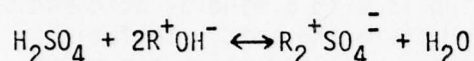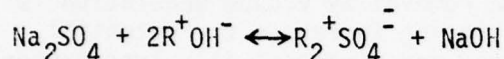
Strong base anion exchange functions well in all pH ranges and is used for water demineralization. Regeneration is with a strong alkali.

d) Weak Base Anion Exchangers. Primary, secondary, and tertiary ammonium groups serve as the functional group or exchange site. They will not split salts and absorb only strong (mineral base) acids. As a result they do not remove carbonic or silica acids, but are more efficient in what they do than the more generalized strong base resins. A typical reaction is:

$$H_2SO_4 + 2R \longleftrightarrow R_2H_2SO_4$$
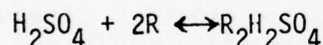
Regeneration is with caustic soda, soda ash (Na_2CO_3), or ammonia (NH_3). Regeneration is more efficient than for strong base resins. In demineralization, they are used in conjunction with strong base resins to reduce regeneration costs and scavenge organics.

3) Other Exchange Operations and Uses. Intermediate acid cation exchangers and intermediate base anion exchangers are sometimes used for ion exchange. Not surprisingly, they are designed to have properties of both strong and weak functional groups.

Intermediate acid cation exchangers have phosphoric acid or some derivative as the functional group and are regenerated with acid. Intermediate base anion exchangers contain both strong and weak base resins. Their behavior is dependent on the strong to weak base resin mix and whether they are regenerated with caustic soda, soda ash, or ammonia.

There are a number of highly porous and adsorbent strong base anion exchangers which can adsorb organic matter such as humic acid and then be regenerated with sodium chloride. Unlike the gelular structure of standard resins, these macroreticular resins have discrete pores and are

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a modification of the original polystyrene divinylbenzene anion exchangers. Such systems are put at the head of a treatment train to protect the demineralizing resins following them.

Ion exchange resins can also be employed as insoluble, inert bases for chemical groups whose specific properties are then used. A common example is removal of dissolved oxygen in water using an anionic resin charged with $SO_3^=$ ions which are oxidized to $SO_4^=$.

Limitations. Ion exchange removes electrolytes from solution. When properly designed, it can remove hardness producing ions (soften), bicarbonate ions (dealkalize), or nearly all ionizable materials (deionize or demineralize) as desired. It is this last operation which is normally desired in high purity water production.

The ion exchange operation will not remove non-electrolytes (with exceptions noted previously). Thus, ion exchange is not designed to remove oils, detergents, many organic compounds, small particulates or microorganisms. These contaminants can foul the ion exchange resin, undermine its effectiveness, and pass through to the product water. When they are present in significant quantities, some form of pretreatment or other high purity water production methods must be considered.

Organic fouling rarely occurs on cation exchange resins but may occur on anion exchangers by organic precipitation. Anion exchange resins attract negatively charged particles which include many organic compounds. Fulvic, tannic, and humic acids are most common. Weak base anion exchangers are more resistant to effects of organic fouling and are sometimes employed as an organic scavenger for the more sensitive strong base anion resins downstream.

Organic precipitation on the resin also serves as a nutrient for microbial growth. Ammonia and nitrates in the water will also support such growth. Although there is no loss in exchange capacity (i.e., no chemical degradation of the resin), microbial growth will result in reduced exchange efficiency and increased pressure drop along the bed. Most importantly it will contaminate product water. Product water quality must be monitored regularly and appropriate control measures taken. For a centralized system this means constant recirculation of water through the resins and periodic chlorination, such as each time the bed is regenerated. Localized systems need to be disinfected regularly by making up some appropriate disinfecting solution and allowing it to sit about an hour in the column. If bacteria overwhelms the resin bed, cleaners and bactericides cannot penetrate the protein sheath and the resin must be replaced.

Colloidal and particulate material can coat ion exchange resins or pass through the resin bed. Pieces of damaged resin may also pass through. When all particulate removal is necessary, ion exchange should be followed by microfiltration. Non-ionic gases such as dissolved oxygen are also not removed by ion exchange.

Iron fouling may occur when insoluble ferric ions coat the surface of resin due to removal by filtration. Another mechanism is to have soluble ferrous ions attach to anion resin sites and be subsequently oxidized to insoluble ferric form, fouling the bed interior. Iron fouling results in reduced bed capacity, inefficient regeneration, and faster resin break-through. It is more likely to occur on anion resins than cation resins, expecially on strong base anion exchangers in the hydroxide form. Pre-treatment considerations include oxidation with subsequent filtration, hot or cold time softening, or ion exchange. Ion exchange for iron removal is normally considered economical when water production rates are less than 400 gallons per minute.

Other water constituents which may foul ion exchange resins when present in sufficient amounts are silica (when greater than approximately 20 mg/l as $CaCO_3$), copper, and aluminum. Pretreatment of these constituents is normally by hot or cold lime softening in large operations or by ion exchange for smaller operations (which is the case for most high purity water systems). Calcium sulfate fouling may occur when a bed containing calcium is regenerated with sulfuric acid and a proper rinse is not executed before placing the bed back into operation. During regeneration the solubility of calcium sulfate is almost always exceeded.

Carbon dioxide affects resin capacity. Strong base anion exchangers can remove CO_2 but consideration should be given to decarbonation if the quantities of CO_2 are large requiring frequent regeneration. Carbon dioxide also lowers the pH of the water which may be undesirable.

Design and Selection Considerations

1) Water Quality. In general, design and selection of the ion exchange treatment operation depends on the quality of the influent and effluent water, the water feed rate, and capital and operational costs. These factors affect the amount and frequency of regenerant dosage and the leakage which can be tolerated compared to the finished quality desired.

Information which should be known about the influent water to the ion exchange unit is summarized in Table 3. Other measurements depend on specific constituents which may interfere with the use of the finished water. More generally, sizing of ion exchange units is especially concerned with the total dissolved solids, total ionizable solids, hardness, and alkalinity.

Leakage is appearance of ions in the effluent which are desired to be removed. Leakage varies with the composition of the influent water and the rate of regeneration of the ion exchange resin. Practically speaking, there is always some leakage. The magnitude of leakage which can be tolerated (the base line leakage) depends on the desired quality of the finished water. Thus, leakage must be measured routinely and provides a convenient way to monitor effluent quality. Table 12 outlines parameters which should be measured routinely in the effluent water according to different ion exchange operations.

TABLE 12. MINIMUM SUGGESTED EFFLUENT WATER QUALITY MEASUREMENTS FOLLOWING ION EXCHANGE

Ion Exchange Operation	Measurement[a]
Strongly acidic cation exchangers, sodium cycle (softening)	Hardness[b]
Strongly acidic cation exchangers, hydrogen cycle (partial deionization)	Acidity,[c] pH,[b] sodium[b] (or hardness)[b]
Weakly acidic cation exchangers (dealkalizing)	Hardness,[b] alkalinity,[b] pH[b]
Strong base anion exchangers, chloride cycle (dealkalizing)	Alkalinity,[b] pH,[b] chlorides[c]
Strong base anion exchangers, hydroxide cycle (partial deionization)	Acidity,[b] pH,[c] silica[b]
Weak base anion exchangers (absorption of strong acids)	Acidity,[b] pH[c]
Cation-anion exchange (deionization or demineralization)	Conductivity,[b] TDS[b]

a. Additional specific measurements of effluent water quality are a function of its intended use.
b. Increases as resin is depleted.
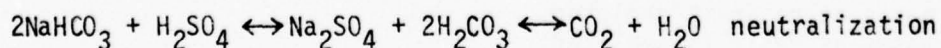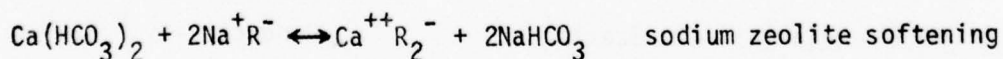c. Decreases as resin is depleted.

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2) Equipment Operation and Configuration. An objective of equipment operation is to operate such that regenerant utilization is a minimum. Common operation is semi-batch, concurrent operation. Water continuously flows down through stationary resins. Regeneration of the resins, also downflow, is at specified intervals. A variation of this is the counter-current technique which employs downflow service and upflow regeneration (or vice versa). In continuous operation, part of the resin is always moving out in frequent ("continuous") pulses to be regenerated elsewhere while regenerated resin is continuously pulsed into the ion exchange vessel. Continuous systems give increased regenerant utilization over fixed resin beds and require reduced resin inventories.

Equipment configurations depend on the treatment desired. Several ion exchange processes have been developed to fill the most common requirements and are discussed below.

a) Sodium zeolite softening. Sodium zeolite softening employs the strongly acidic cation exchangers in the sodium cycle. Although higher capacity organic resins have replaced the zeolite, they are still referred to as zeolite softeners. Operation of zeolite softeners is simple and efficient. Variations in raw water flow rate have little effect. Zeolite softeners have a low investment cost and lend themselves to easy automatic control. Regeneration is normally with a 10 percent salt solution.

b) Softening and dealkalization: sodium zeolite - hydrogen zeolite split stream. In this type of treatment, incoming source water is split. Part of the water goes through a strongly acidic cation exchanger in the sodium cycle and the remainder goes through a strongly acidic exchanger in the hydrogen cycle. The sodium zeolite exchanger efficiently softens the water. The hydrogen zeolite exchanger softens and produces mineral acids to neutralize the alkalinity in the water. Alkalinity is converted to unstable carbonic acid which is easily removed by degasification. Typical reactions are:

$$Ca(HCO_3)_2 + 2Na^+R^- \longleftrightarrow Ca^{++}R_2^- + 2NaHCO_3 \qquad \text{sodium zeolite softening}$$

$$MgSO_4 + 2H^+R^- \longleftrightarrow Mg^{++}R_2^- + H_2SO_4 \qquad \text{hydrogen zeolite softening}$$

$$2NaHCO_3 + H_2SO_4 \longleftrightarrow Na_2SO_4 + 2H_2CO_3 \longleftrightarrow CO_2 + H_2O \quad \text{neutralization}$$

Desired alkalinity of the treated water is maintained by varying the percentage of sodium and hydrogen treated water in the blend.

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c) Softening and dealkalization: sodium zeolite - chloride anion dealkalization. Influent water goes through a strongly acidic exchanger in the sodium cycle to remove hardness, then through a strong base anion exchanger in the chloride cycle to remove alkalinity. Typical reactions are:

$$Ca(HCO_3)_2 + 2Na^+R^- \longleftrightarrow Ca^{++}R_2^- + 2NaHCO_3$$

$$NaHCO_3 + 2R^+Cl^- \longleftrightarrow NaCl + R^+HCO_3^-$$
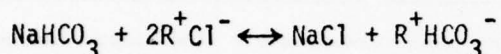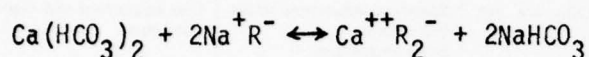
Initial costs of this type of softening/dealkalization are higher than sodium-hydrogen zeolite split stream softening/dealkalization. However, the process involves no handling of acids and readily lends itself to automatic control.
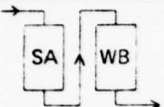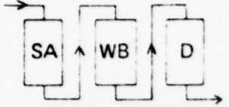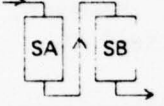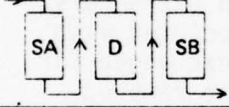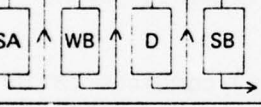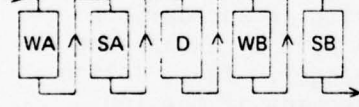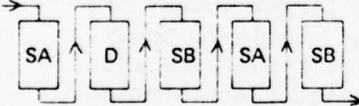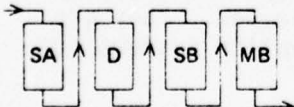
d) Softening and dealkalization: weakly acidic cation exchangers. This process involves only calcium and magnesium ions associated with alkalinity and was illustrated earlier. The cations are exchanged for hydrogen ions. The resulting unstable carbonic acid can be removed by degasification. Best results are found with hard, alkaline, low sodium waters.

e) Demineralization. Demineralization (deionization) combines cation and anion removal and is frequently used synonymously with ion exchange. Demineralization changes metal salts to their acids with an appropriate cation (usually hydrogen) exchanger, then removes the acid by exchanging it with an appropriate anion (usually hydroxide) exchanger. Thus, cation removal is always followed by anion removal and may use any of the ion exchange operations discussed above. In general, the larger number of beds in series, the better quality of water resulting. Table 13 shows some typical demineralization treatment trains using multiple beds and also lists appropriate comments. It should also be noted that selected resins can remove carbon dioxide, chlorine, ammonia, and other ionizable gases and act as pre- or post-treatment operations.

Table 13 also illustrates the use of mixed beds. Mixed beds combine cation and anion resins in a single vessel. For small operations, mixed beds are economically attractive and no deaeration is needed. They are frequently used as a polisher following primary deionization systems but may be used in lieu of multiple bed exchangers.

Mixed beds act like an infinite number of cation and anion exchangers in series. As a result, they are capable of producing a finished water of high ionic purity and near neutral pH. Quality of the finished water remains constant throughout the cycle. Regeneration of the mixed bed requires segregation of the anions and cations followed by remixing with compressed air.

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TABLE 13. TYPICAL DEMINERALIZATION TREATMENT TRAINS

DEMINERALIZER SYSTEM	APPLICATION	TYPICAL EFFLUENT	ADVANTAGES AND DISADVANTAGES
SA → WB	Silica and CO_2 are not objectionable.	Specific conductance 10-30 micromhos, Silica unchanged.	Low equipment and regenerant costs.
SA → WB → D	Silica is not objectionable but CO_2 removal is required.	Specific conductance 10-20 micromhos, Silica unchanged.	Low regenerant costs, but requires repumping.
SA → SB	Low alkalinity raw water, silica removal required.	Specific conductance 5-15 micromhos, Silica 0.02 to 0.10 ppm.	Low equipment costs, repumping not required, high chemical costs.
SA → D → SB	High alkalinity, raw water, silica removal required.	Specific conductance 5-15 micromhos, Silica 0.02 to 0.10 ppm.	Low chemical costs, repumping is required.
SA → WB → D → SB	High alkalinity, sulfate and chloride raw water. Silica removal required.	Specific conductance 5-15 micromhos, Silica 0.02 to 0.10 ppm.	Low chemical costs, high equipment costs, repumping required.
WA → SA → D → WB → SB	High hardness, alkalinity, sulfate and chloride raw water. Silica removal required.	Specific conductance 5-15 micromhos, Silica 0.02 to 0.10 ppm.	Lowest chemical cost, high equipment cost, repumping required.
SA → D → SB → SA → SB	High alkalinity, high sodium raw water, high purity treated water required.	Specific conductance 1-5 micromhos, Silica 0.01 to 0.05 ppm.	Low chemical costs, high equipment costs, repumping required.
MB	Low solids, raw water, high purity treated water required.	Specific conductance is less than 1 micromho, Silica 0.01 to 0.05 ppm.	Low equipment cost, high chemical cost.
SA → D → SB → MB	High alkalinity and dissolved solids raw water, high purity treated water required.	Specific conductance is less than 1 micromho, Silica 0.01 to 0.05 ppm.	Lower chemical cost, higher equipment cost, requires repumping.

KEY

SA STRONGLY ACIDIC HYDROGEN CATION	**WA** WEAKLY ACIDIC HYDROGEN CATION	**WB** WEAK BASE ANION
SB STRONG BASE ANION	**D** DEGASIFIER OR VACUUM DEAERATOR	**MB** MIXED BED

Source: "Betz Handbook of Industrial Water Conditioning," 6th Betz Laboratories, Inc., Trevose, PA (1976).

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The cost of ion exchange operations is roughly proportional to the concentration of ionized impurities. Generally, regeneration is more economical than replacement if the pure water production is expected to be greater than 1 gpm. If less, it is advantageous to use disposable cartridges. When total dissolved solids of the influent water exceed 1000 mg/l or if total ionizable solids exceed 300 mg/l, pretreatment by reverse osmosis or treatment alternatives such as distillation must be considered.

Department of the Army Technical Manual (TM) 5-813-3 Water Supply, Water Treatment discusses criteria to be followed in determining the necessity for and extent of treatment at Army installation and references related publications on the subject.

Reverse Osmosis

Description. Reverse osmosis is a membrane separation operation in which water molecules pass through a semi-permeable membrane but solute molecules are retained. Reverse osmosis operation depends on a pressure driving force to overcome the natural osmotic pressure caused by the dissolved salts in the water. With existing membranes and equipment, operating pressures vary from atmospheric to 1500 psig. Typical operating pressures are in the neighborhood of 600 psig.

Salt rejection by reverse osmosis membranes is a physical-chemical phenomenon which involves repulsion of ions from the surface of the membrane and adsorption and subsequent diffusion of pure water through the membrane. Higher valence ions are repelled furthest away from the membrane. Thus, divalent ions such as calcium are rejected somewhat better than monovalent ions like sodium. Generally, salt rejection by reverse osmosis may vary from 85 to over 95 percent.

Neutral organics in the wastewater have no charge and therefore there is no electrical interaction between them and the membrane. Rejection of such organics by reverse osmosis molecules is based on a sieve mechanism and is related to the size and shape of the organic molecule. Organic molecules tend to lower the interfacial tension between the solution and the membrane. This allows lower molecular weight molecules to pass through membrane pores which are about 0.02 μm to 0.05 μm wide and a function of feed flow and pressure. Organic molecules having molecular weights less than approximately 200 can be expected to pass through the membrane. Molecules with higher weights can be expected to be rejected. Thus, low molecular weight alcohols, fatty acids, phenolic compounds and formaldehyde will pass through; sugars, bacteria, pyrogens and viruses are rejected. Organic rejection will vary from 0 to 100 percent depending on the size of the organic compound and upon the degree of ionization of the compound (especially in the case of organic acids and bases).

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Incorporation of membranes into effective hardware has resulted in various types of equipment classified as follows:

1) Spiral Wound. A porous membrane is folded over a porous incompressible backing material and wrapped around a water takeoff tube. This assembly is then placed inside a pressure vessel.

2) Tubular Modules. Membranes are formed in the shape of a cylinder and inserted into porous fiber glass reinforced epoxy tubes approximately one-half inch in diameter.

3) Multiple Plate. Membranes are placed in a unit similar to a plate and frame filter press.

4) Hollow Fiber. Hollow fibers 50 µm O.D. and 25 µm I.D. are packed into a cylindrical shell resembling a tube heat exchanger. The input water, under high pressure, flows over the outside surface. Treated water (permeate) is collected inside the fiber bore. Concentrated organics remain outside and are removed as brine (concentrate).

Cellulose acetate is the most common membrane material. Hollow fiber reverse osmosis configurations use an aromatic polyamide material related to nylon as the membrane material.

Limitations. Cellulose acetate membranes are pH sensitive. Acetate radicals will be quickly hydrolyzed outside a pH range of approximately 4-8 giving straight cellulose which has no salt rejection capacity. Polyamide fibers are stable over a wider pH range. Polyamide membranes, however, will degrade in the presence of chlorine and cannot be used to treat waters having chlorine residuals.

Membranes soften as temperature increases. Above approximately 100°F, membrane compaction rates become significant and flux decline occurs. High operating pressures, especially above 500 psig, also contribute to gradual membrane compaction and flux decline.

Reverse osmosis membranes are expendable. Hydrolysis, flux decline, and physical weakening make it necessary to replace membranes periodically. Cellulose acetate membranes are usually replaced every 2-3 years. Polyamide fibers, with their superior chemical and physical properties, are usually replaced every 3-5 years.

Like other unit operations, large concentrations of suspended solids may foul the membrane filters. Spool wound fiber or paper cartridge filters are almost always used before the membrane filter and are designed to remove particles larger than 5-25 µm from the influent water. Colloidal turbidity may have to be removed by filtration assisted by coagulation/flocculation.

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Bacteria concentrations must be controlled. Cellulose acetate is fairly resistant to bacterial attack, but some strains can actually digest the cellulose acetate reducing its salt rejection capacity. Polyamide fibers are more resistant to bacterial attack. Large growths of bacteria can impair flow. Microorganisms may be controlled by maintaining a constant chlorine (for cellulose acetate) or formaldehyde concentration in the feed. Formaldehyde sterilization is desirable because it can pass through the membrane and sterilize both sides.

Reverse osmosis membranes do not reject non-ionized forms of gases well. Examples of such gases commonly found in water or wastewater are oxygen, carbon dioxide, and hydrogen sulfide.

Precipitation of calcium carbonate can foul the membrane. This problem is normally taken care of by maintaining pH between 5 and 6 which promotes bicarbonate formation from the carbonate ion. Calcium sulfate causes similar problems but pretreatment is more difficult because the solubility is relatively unaffected by pH changes. When relatively large quantities of hardness producing anions or cations not amenable to pH control are present, other means of softening must be considered. This includes hot and cold lime softening, ion exchange, and chelation or sequestration.

Iron and manganese behave similarly to hardness producing ions and in adequate amounts can cause serious fouling. Precipitation occurs upon oxidation of their soluble (OUS) forms to higher insoluble oxidation states of hydrous ferric and manganese oxides. Usual pretreatment is to oxidize them to insoluble form and then filter them out. An alternative is to reduce existing insoluble ions to (ous) forms with a reducing agent such as sulfite and keep them in solution. Deoxygenation of the influent can be employed to prevent re-oxidation.

Precipitation is a reversible limitation of reverse osmosis membranes. Deposits can usually be removed with a cleaning compound which acts within the pH limits of the membrane (caustic cleaning agents cannot be used). Surface coating of the membrane pores by oil is also reversible. For such cases an oil dispersion agent must be used.

It is important to recognize the practical aspects of reverse osmosis equipment. Pinholes may develop in the spiral wound membrane sheets. Small leaks may appear in the glued seams of the seals. Some of the million or so hollow fibers may break in their cylindrical shell. For reverse osmosis membranes to work perfectly they must be formed and packaged perfectly. Thus, actual treatment efficiency will be less than potential treatment efficiency because of mechanical imperfections. For example, it may be expected that some bacteria, pyrogens or virus will get through the membrane. If biologically pure water is required, distillation, microfiltration, or sterilization including electronic (ultraviolet) water sterilization should be considered for post RO treatment.

Design and Selection Considerations

1) Water Quality. Table 3 outlines suggested influent water quality analyses. Removal of wastewater constituents which may lower membrane flow-through efficiency has already been discussed. Potential contaminants were suspended and colloidal solids which may coat and foul the membrane and scale-producing ions which may precipitate out. As influent water quality is improved, time increases between cleaning cycles. Water production and water quality improves. The decision about the extent of pretreatment is dependent on its cost compared to the benefits gained. Pre-filters to remove most particulate matter are commonly installed before membrane separation at the beginning of the treatment train.

Effluent water quality is commonly measured with a conductivity meter which measures ionic purity. Additional specific contaminants are measured as required according to product water use.

2) Equipment Operation and Configuration. The most successful membrane equipment configurations for commercial purposes are spiral wound and hollow fiber. Spiral wound cellulose acetate membranes have a high membrane surface area per unit volume ratio which is desirable. They are usually installed in series in their pressure housing. Spaces between membrane sheets are large enough to allow membranes to be readily cleaned when necessary. Flow through the filter is relatively turbulent and can dislodge algae which may grow on the membrane. Influent water should be filtered through a 25 micron cartridge filter for best performance. The cellulose acetate membrane can tolerate a continuous chlorine feed for disinfection. Normal operation should be at pH 5.5-6.0 to avoid hydrolyses of the cellulose acetate and precipitation of calcium carbonate.

Hollow fiber aromatic polyamide membranes have greater chemical and physical stability than cellulose acetate membranes with one exception; they cannot tolerate a chlorine content in the water. They will degrade when total chlorine content is equal to or greater than 0.05 ppm. Thus, one must use other bactericides than chlorine to prevent bacterial growth. They cannot be attached directly to tap water outlets which provide water with a chlorine residual. Flow through the filter is laminar to avoid breaking fibers and therefore they are more susceptible to fouling. Influent water should be filtered through a 5 micron filter. Normal operation should be at pH 5.5-6.0 to avoid precipitation or calcium carbonate. Cost of water purification is about the same for hollow fiber and spiral wound membrane equipment.

Operational monitoring of the effluent from the reverse osmosis operation is usually done by noting fall off in the permeate rate at constant pressure. When permeate rates become unacceptable low -- or conversely when the pressure drop becomes unacceptably high to maintain a constant permeate rate -- the membrane must be cleaned with a detergent.

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Sudden increases in contaminants suggest leaks. One way to test for leaks is to add food dye to the influent water. If no pin holes, broken seals or broken fibers exist the dye will not come through.

Distillation

Description. Distillation removes water from impurities by converting the water to vapor, then recondensing it as distilled water. This action is the reverse of other water purification methods (which remove impurities from the water) and requires considerable energy.

Energy input to stills can be steam, electricity, or gas. Gas fired stills are seldom used because of their complicated design. Stills producing greater than 10 gph of distillate are normally heated by steam because of the cost of electricity.

Properly designed stills can produce water free of bacteria, virus, and pyrogens. Impurity volatilization and entrainment are the biggest treatment limitations distillation faces. Like all treatment methods, the distillate produced from a still will vary with the quality of the feedwater. In addition, the condition and maintenance of the still plays a major part in the quality of the distillate.

There are several general types of stills:

1) Single Effect Stills. Such stills produce a distillate resulting from a single phase change. A typical example is the conventional laboratory still, electrically operated, and producing anywhere from 1/2 to 10 gph of distillate. Total dissolved solids of the distillate is normally <1 mg/l, pH varies 5.4-7.2, and resistivity is in the 0.3 to 0.8 megohm-cm range.

2) Multiple Effect Stills. These are several stills in parallel where the steam produced by the first is used to evaporate water in the second; the resultant steam from the second boils water in a third and so on. Multiple effect stills take advantage of the latent heat of steam from one still to preheat incoming feed to the next. They are operated under pressure or vacuum so that there is a slight temperature drop going from one effect to another. Initial capital costs for multiple effect stills are higher than conventional single effect stills but operating costs diminish in proportion to the number of effects. Multiple effect stills can produce water having a resistivity in the range of 0.8-1.0 megohms-cm.

3) Multiple Stills. These are stills in series in which the distillate from the first still serves as the feedwater to the second; the distillate from the second is fed to the third and so on. Stills with glass redistillers would also fall in this category. The cost to produce water from such stills increases in proportion to the number of stills. Multiple stills are used when very high purity water including pyrogen-free water is needed. Ionic purity of water from such stills will not be

extremely high, however, because of volatile ionizable gases such as ammonia and carbon dioxide which are vaporized, then redissolved in the condensate. Additional treatment such as degasification or ion exchange is usually employed to remove such gases if desired. Resistivity of water produced from multiple stills can be as high as 1-2 megohms-cm.

4) Other Distillation Methods. Other distillation methods are generally more complex but require less energy for operation. Included in this category are vapor compression stills, centrifugal rotating heat exchanger stills, and falling film evaporator stills.

Limitations. Vaporation of impurities with boiling points close to or below that of water is an inherent disadvantage of the distillation process. Such impurities, usually dissolved gases, are vaporized, then redissolved in the condensate. Ammonia, chlorine, and carbon dioxide are typical examples of such gases. If presence of such gases is unwanted, pretreatment by ion exchange or deaeration is necessary. Ion exchange will only remove ionizable gases (ammonia, chlorine and carbon dioxide are ionizable gases in water). Filming and neutralizing (soluble) amines used in boiler water and steam treatment for corrosion control also will contaminate distillate if the steam produced by such boilers is used as a feed to the still or as a distillate scrubber.

Entrainment and carry-over of unvaporized droplets or small particles (<1000 μm) in the vapor are potential sources of contamination. Scale formation is a problem and proper measures must be taken depending on the quality of the incoming water and desired quality of the product water. Sodium cycle cation exchangers will remove the Ca^{++} and Mg^{++} hardness producing cations and exchange them for sodium (which is acceptable only if high levels of sodium can be tolerated in the finished product). An alternative is to pass part of the influent water through a hydrogen cycle cation exchanger. This is desirable when the influent water has a significant alkalinity content and relatively low hardness content. The resulting mineral acids from the hydrogen cycle ion exchange process can neutralize the alkalinity in the untreated water resulting in a final water having a reduced hardness and alkalinity going into the still.

Scaling and corrosion problems in distillation units are similar to those encountered for low pressure boilers. The section on water treatment in Department of the Army Technical Manual (TM) 5-650, Central Boiler Plants, gives a good background discussion about water impurities affecting boiler operations and gives examples of common treatment methods to remove them. Boiler water systems are also fully discussed in the "Betz Handbook of Industrial Water Conditioning."

As a single unit operation and for small volume localized use, a still is a practical source of high purity water. There are several factors against its use as the sole treatment for a large volume operation. High

volume production equipment is massive and uses considerable energy. Plumbing and maintenance is relatively complicated. Quality of distilled water depends to a large degree on proper operating procedure, maintenance scheduling, and operator vigilance. Energy costs for redistillation lend distillation to be associated closely with storage of produced high purity water rather than recirculation and retreatment of the water in a closed loop system.

Design and Selection Considerations

1) Water Quality. Feedwater hardness is important because calcium and magnesium scale forming deposits affect the efficiency and maintainability of the distillation unit. Similarly, feedwater alkalinity usually in bicarbonate form can combine with calcium and magnesium to form scales or, upon heating result in carbon dioxide which may be later absorbed in the distillate forming corrosive carbonic acid. Dissolved oxygen also promotes the rate of corrosion. Table 3 outlines a suggested influent water quality analyses. Table 14 suggests water softening criteria. Hardness of less than 50 mg/l as $CaCO_3$ is desirable in still feedwater.

TABLE 14. WATER SOFTENING CRITERIA

A. GENERAL

0-75 mg/l total hardness as $CaCO_3$ = soft water
75-150 mg/l total hardness as $CaCO_3$ = moderately hard water
150-300 mg/l total hardness as $CaCO_3$ = hard water
>300 mg/l total hardness as $CaCO_3$ = very hard water

B. ARMY GUIDELINES

Hospital Water. Soften entire hospital water supply when hardness exceeds 171 mg/l as $CaCO_3$. Blending a zero hardness treated high purity water with the influent water to a hardness of approximately 51 mg/l as $CaCO_3$ is recommended. Special studies may justify additional softening.

Laundry Water. Soften influent water when hardness exceeds 43 mg/l as $CaCO_3$. Softening the influent water to zero hardness is recommended.

Mess Hall Water. Installation of softeners for small mess halls is not recommended. Softening influent water to 43 mg/l as $CaCO_3$ for large central mess halls may be justified to protect equipment and insure satisfactory washing of dishes.

Source: Department of the Army Technical Manual 5-813-3, "Water Supply, Water Treatment" (Sep 1966).

Required quality of the finished water is, of course, very important when considering distillation for high purity water treatment. Distillation can produce biologically pure and pyrogen-free water unaided, a claim no other single unit operation can make. On the other hand, distillation is only a fair demineralizer and one must take into account potential contamination from volatile impurities and carry-over. Such impurities, however, are only significant if they exist in the feed water and cannot be tolerated in the finished water. Still designs take into consideration the limitations of the distillation operation and try to minimize their effects as is detailed in the next section.

2) Equipment Operation and Configuration. The following features are either built in or are options to stills:

a) Baffles. Reduce water droplet and pyrogen carryover. There are several configurations depending on the purity of water desired.

b) High Vapor Disengaging Space. An extra high evaporator space to reduce the possibility of carryover by foaming, splashing, or priming as the water is boiled.

c) Inclined Condenser. Promotes venting to the atmosphere of unwanted volatile gases and minimizes impurity reabsorption.

d) Constant Bleeder. Is necessary to deconcentrate impurities left in the evaporator and retard scale formation.

e) Countercurrent Condenser. Uses steam produced for condensation to preheat feed water and reduce fuel costs.

f) Thermal Control. Allows gentle boiling which minimizes carryover from splashing and priming.

g) Distillate Coolers. Additional coils quickly cool distillate to temperatures suitable for use (for example, feed to ion exchange units).

h) Steam Scrubbing Chambers. Purified steam runs countercurrent to the distillate promoting transfer of impurities from the distillate to the steam.

i) Cyclone Chambers. Water vapor is directed in a spiral motion and microscopic liquid and solid particles are separated by centrifugal force.

Stills heated by steam require steam pressures of 35-60 psig of saturated steam. Nine pounds of steam at 50 psig and 9 gallons of cooling water are required to produce a gallon of distillate. Nine hundred and seventy BTU's of heat are needed to change 1 pound of water to steam. Electrically operated stills use about 2.6 kw per gallon of distillate.

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Unlike ion exchange or reverse osmosis, the cost of water production varies less with influent water quality and more with the degree of maintenance of the equipment.

Filtration

Description. Filtration is the process of passing a liquid through a filtering medium for the removal of suspended or colloidal matter. Filtration may be employed to pre-filter and protect other high purity water treatment operations downstream. It may be employed as an independent unit operation designed for particulate removal. In such cases it is situated at the end of the treatment train just prior to high purity water use.

Pre-filters used upstream to filter out relatively large material and remove the bulk of the solids are also referred to as coarse filters, depth filters and raw water filters. Such filters may use granular material, finely woven cloth, unglazed procelain, or porous paper membranes depending on the quality of the influent water. In these filters, particles or solids greater than about 10 microns are separated from solution by a physical sieving action.

Other types of pre-filters use different mechanisms for solids separation. Macroreticular resins are a synthetic hybrid between carbon and ion exchange. Resin beads have pores like carbon and a weak ion exchange capacity. Macroreticular resin beds work well in removing colloids in the 0.001-0.5 micron range which may plug microfilters. (Macroreticular resins were discussed earlier as a pretreatment to eliminate organic fouling of ion-exchange resins).

In high purity water production, filtration normally refers to microscopic filtration of extremely fine particles, usually 10 microns or less in size. Microfiltration is accomplished by passing the solution through a polymeric membrane sheet having regularly-spaced, precisely-sized holes or pores. When the pores of the membrane filter are 1.2 microns or less in diameter, the filtering process is frequently termed submicron filtration or absolute filtration.

Microfilters commonly use membranes having pore sizes of 1.2, 0.45, or 0.22 microns in diameter. For dry sterilization, membranes having pore sizes 0.45 are used since most known bacteria are in the 0.5-1 micron size range. Microfilter membranes with pore diameters of 0.45 microns can filter out staphylococci, pseudomonads, and fecal coliforms. Membrane filters with 0.22 micron pores may follow the 0.45 micron pore filters as "insurance."

Most common microfilters have a plate and frame type filter press configuration. They are designed for localized high purity water systems and because of their filtering configuration (flow is perpendicular to

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the filter surface) they are not practical for filtering large water volumes because of potential clogging. Tubular type microfiltration systems are designed to handle relatively large water flows (50-100 gallons per square foot of membrane area per day) at low pressures (5-50 psig). They employ microporous tubes made of inert thermoplastic materials. Water flow is across rather than normal to the membrane surface and subsequent scouring allows them to handle influent waters with higher suspended solid content. Controlled porosity of the tubular membranes along with tube length and tube grouping selection (to form a multi-tubular module) allows tubular microfilters to be tailored to specific situations or effluents.

Ultrafiltration is also a membrane filtration process which selectively filters out particulates. Ultrafiltration (as well as microfiltration) is similar to reverse osmosis in that it is a pressure driven membrane process. Unlike reverse osmosis, the predominant separation mechanism is a physical-sieving action rather than solution-diffusion mechanism. As a result, osmotic pressure is negligible and typical operating pressures are much lower.

Common ultrafiltration equipment configurations are hollow fiber, tubular, or spiral wound. Ultrafiltration produces a finer sieving action than microfiltration and can also reject macrosolutes of molecular weight above approximately 500 or higher, depending on the membrane selected.

Typical operating pressures range 10-100 psig and throughput or flux is 20-50 gallons per square foot per day. Table 15 illustrates typical ranges of particles removed for various processes including those discussed. These ranges represent potential particle sizes which can be removed by the various unit processes. The information in Table 15 is best interpreted in relative terms rather than in absolute sizes which may vary somewhat. It is also important to keep in mind the limitations which were discussed for the various unit processes and to remember that filtration is only one of the mechanisms used for particle separation.

Limitations. Water to be filtered must be relatively clean. Influent quality, of course, depends on the type of filtration to be employed. Microfilters can be easily plugged by colloidal or gelatinous type material which approach pore size. As a result, pre-filters are commonly employed.

Membrane filters, whether in microfilters or ultrafilters are subject to tears or imperfections. Thus, actual treatment efficiency may be less than design efficiency because of mechanical deficiencies. Pore sizes for microfilters also behave like a sponge and may vary with the pressure and rate of water flow through them. Similarly, ultrafiltration of large molecules is influenced by the molecules' cross-section or shape which bears no fixed relationship to molecular weight except, perhaps, in

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TABLE 15.   PARTICLE REMOVAL PROCESSES

Process	Particle Sizes Removed
Reverse Osmosis	
Dialysis	
Electrodialysis	
Ion Exchange	
Distillation	
Freezing	
Solvent Extraction	
Ultrafiltration	
Ultracentrifuges	
Microfilters	
Centrifuges	
Bubble Fractionation	
Cyclone Separators	
Sedimentation	
Cloth Filters	
Screening	

Angstroms	1	10	10^2	10^3	10^4	10^5	10^6	10^7
Millimicrons	10^{-1}	1	10	10^2	10^3	10^4	10^5	10^6
Microns	10^{-4}	10^{-3}	10^{-2}	10^{-1}	1	10	10^2	10^3
Millimeters	10^{-7}	10^{-6}	10^{-5}	10^{-4}	10^{-3}	10^{-2}	10^{-1}	1

Source: "Reverse Osmosis, Ultrafiltration and Eastman
Membrane," Eastman Chemical Products, Inc.,
Kingsport, TN (1971).

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a homologous series. Ultrafiltration is also subject to fouling from precipitation as well as other membrane limitations discussed under reverse osmosis. Not surprisingly, membranes with the smallest pores are most sensitive to fouling and clogging while those with larger pores are less sensitive.

When using microfilters or ultrafilters as bacteria or particulate filters for other than water, the filters efficiency may be a hidden liability. For instance, when dry sterilizing heat-liable medicaments in the pharmacy, the filter may retain part of the drug. For such cases, one should use the coarsest filter which will effectively filter out the desired particulates.

Design and Selection Considerations. Prefilters are employed for initial removal of suspended solids or colloidal material and serve to protect downstream operations. Prefilters which remove suspended solids are normally used to pretreat incoming waters of other than potable quality. They are placed at the beginning of the treatment train. Prefilters such as macroreticular resins which are designed to remove colloidal material and some organic matter are placed just before the treatment operation they are designed to protect. Normally they will protect ion exchange resins or microfilters.

Microfilters are commonly placed just prior to use of the water. They serve to filter particulates (including particulates as ion exchange resin fragments) and bacteria. In industrial situations, microfilters may be used as a pre-filter and placed before the reverse osmosis operation. Design of microfilters takes into consideration expected operating temperatures, pressures, pore size distribution and feed circulation velocity. Tubular type microfilters made of thermoplastic materials are usually inert to the chemical properties of the feed water.

The design parameters and approach for ultrafiltration design are quite similar to those for reverse osmosis. One major difference is that concentration polarization plays a much larger role in ultrafiltration. Concentration polarization is a boundary layer effect which affects flux across the membrane and is a result of rejected species building up at the membrane surface. It is a much more significant problem in an industrial or reuse situation where concentrations of unwanted species are high, rather than for high purity water production where the relative quality of the feed water is much better.

Another important difference between ultrafiltration and reverse osmosis membranes is their chemical composition. The chemical composition of reverse osmosis membranes is of primary importance in determining the rejection of solutes. The chemical composition of ultrafiltration membranes is not and, therefore, its membranes can be designed to withstand greater extremes of temperature, pH, and oxidants.

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Carbon Adsorption

Description. Activated carbon removes contaminants by adsorption or attraction of them to its surface. It is commonly used to remove residual chlorine and organic compounds. A secondary function of activated carbon filtration is mechanical filtration.

Activated carbon's large surface area and its pore structure are more important than its chemical characteristics. Molecules close to the carbons pore size are attracted the most. Typical surface areas for granular carbon vary 500-1400 m^2/gm of carbon. Thus, external carbon particle size contributes little to total surface area. It will affect adsorption rate but not ultimate adsorption capacity. Because of easier handling characteristics, granular activated carbon is more commonly used than powdered activated carbon.

Limitations. In water, activated carbon has a preference for large organic molecules and for substances which are non-polar in nature. It exhibits poor removal efficiencies for low molecular weight, soluble organics.

Desorption may occur at pH values above nine. At best, adsorption is poor at high pH. Adsorption efficiency also decreases as water temperature decreases.

Carbon beds can become nutrient reserves for bacteria and sources of pyrogens. In extreme instances, some bacteria may even resist the regeneration process to multiply again when the column is returned to service.

Design and Selection Considerations. Carbon column design approaches are well defined and will not be repeated in detail here. The approaches consider the general areas of carbon selection and column size, operation and configuration.

It is worthwhile to note that expanded upflow columns should be selected when concentrations of suspended solids are significant and it is desired to utilize the column's adsorption potential most efficiently. If a dual role of organic removal and suspended solid removal is desired, a downflow column configuration should be employed. It is best, however, to precede carbon adsorption with a mixed media pre-filter when suspended solid concentrations are large enough to potentially impede carbon operation.

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Carbon adsorption involves transporting the solute to the carbon exterior, diffusing the solute throughout the pores, and finally adsorbing the solute on the pore surfaces. Hydraulic loading rate does affect adsorption efficiency. If it is too slow, the solute content of the liquid film by the particle is depleted faster than it is replaced. If it is too fast, the carbon cannot remove the solute as fast as it is transported to its surface. Typical hydraulic loading ranges from 2 to 10 gpm per square foot of column cross sectional area.

General wastewater quality parameters of concern are suspended solids, organic content (as BOD, COD, TOC, or possibly MBAS for detergent removal), pH, and temperature. Specific wastewater quality concerns depend on the finished water use and often include chlorine and phenols. In practice, suspended solids are a concern because they increase the headloss through the column requiring more frequent backwashing.

Total organic carbon content is the quickest, most reliable way to measure activated carbon performance. In high purity water, when organic contents approach zero, the preciseness of this test becomes limiting. Other more specific tests must be considered which unfortunately may involve detailed and time consuming analytical procedures. As a supplement to regular water quality testing (which for some constituents may not be as frequent as desirable), carbon should be exchanged on a regular, frequent basis (for example, each time membrane filters are replaced).

Regenerative organic removal filters are mixtures of activated carbon and a special regenerative adsorbent anionic resin. The resins can be regenerated with sodium hydroxide. Contents of the adsorbent mixture contained in these filters are normally patented by the producer.

DISTRIBUTION AND STORAGE

General

It is inconsonant that a most desirable characteristic of high purity water, its ability as an excellent solvent, should be so undesirable with respect to storage and distribution. Once produced, the quality of high purity water begins to deteriorate.

The atmosphere, container, distribution system, and personnel in contact with the high purity water ultimately degrade its quality over time. As such, a goal of all storage and distribution systems should be to get the freshly produced high purity water to the user as fast as quality requirements dictate and economic constraints will allow.

Practically speaking, it is not always possible to route freshly produced high purity water direct to the user. Preventive measures must be taken to minimize water contamination from the time the high purity water

is produced until the time it is used. This includes selection of proper materials for the storage and distribution facilities, judicious routing of the high purity water, and (for storage) protection from the environment. These topics are discussed below.

Materials of Construction

Tin. Pure tin, also called block tin, is widely considered to be the most inert material to high purity water. Tin is too soft to be used alone and tin lined pipe is used for distribution systems. Pipe backing is usually copper or brass but may be bronze, nickle or steel. Tin-lined pipe has a 1/16-1/8 inch coating of tin. Tin-coated pipe has a much thinner coating and is not as durable. The condenser portion of stills and storage tanks may also be lined with tin or a silver-tin alloy.

Although the most desirable material from a water quality standpoint, cost and installation of tin-lined piping is expensive. Special tools and trained personnel are required to insure proper pipe installation. Very clean working conditions and careful attention must be paid to joining pipe fittings and couplings to prevent any interruption in the pipe lining or contamination from pipe threading oils. In this respect, Teflon tape and/or Teflon coated O-rings may be used during installation.

Because tin-lined or tin-coated pipe installation requires special care, it is desirable to require the installer to furnish a guarantee as to the integrity of the distribution system. Regular monitoring for copper (at the ppb level) will signal failure of the tin lining and contamination of the distribution system when copper, bronze, or brass is used as the base support. Iron, aluminum, lead, zinc, or total heavy metals may also be monitored as conditions dictate.

Plastics. Plastic materials come closest to rivaling tin as an inert material desirable for high purity water distribution. In general, plastics are less expensive than tin, much easier to install, and more commonly used than tin in distribution systems. Unfortunately, diameters of 3/4 inch or larger may not be available for some plastic materials, limiting them to local distribution systems. All plastics have the potential of leaching organic and inorganic contamination from vessels from which they were manufactured. Flushing the distribution system with an acid solution as a pretreatment before initial use is desirable to leach manufacturing contaminants out.

Polytetrafluoroethylene (often abbreviated TFE or referred to by the brand name Teflon) is a desirable material for high purity water distribution. It is an inert hydrophobic material which will not contaminate the water. Teflon flexible tubing is available which has an inner layer of pure Teflon, a middle layer of tight fiberglass yarn braid, and an outer layer latex rubber sheath or neoprene. Since it is flexible, elbows are not needed and tubing may be easily joined using rigid Teflon inserts,

tees, and conventional hose clamps. Disadvantages of Teflon are that it is expensive and will experience cold flow under pressure. Joints might leak after several months of installation.

Teflon lined pipe is another alternative and can be manufactured in larger diameters than tubing. Care must be taken to insure the lining is of pure Teflon and not merely a coating which has been sprayed on. Teflon lined pipes and fittings are excellent for the distribution of pure water. However, the system is expensive and, as with tin lined pipe, must be carefully checked for continuity after installation. The Society of the Plastics Industry, Inc., has promulgated standards and specifications for this type of pipe.

After Teflon, polypropylene is desirable for high purity water use. Polypropylene is manufactured as tubes, pipes, and containers and will maintain its form in high temperatures (to 230°F). Polyethylene is similar to polypropylene but will distort at temperatures above 140°F. The plastic carboys commonly used to store distilled water are usually of polyethylene. Polyethylene is porous and carbon dioxide may penetrate the storage container, lowering its ionic purity.

Polyvinylchloride (PVC) is available in many formulations and is probably the most widely used material for high purity water distribution. Like polyethylene it will distort at temperatures above 140°F. It also has more of a tendency to shed plasticizers and filler materials than most other plastics and as a result is probably the least desirable of the common plastic materials used.

Rubber and similar materials including Tygon can be a source of contamination. Pure water may attack some of these substances such that discoloration and cracking will occur within a few months.

Glass. Hard borosilicate glass is used to make glass pipe and glass lined vessels. The use of glass for high purity water storage and distribution is particularly prevalent in the pharmaceutical industry. Borosilicate glass is relatively soluble in high purity water and at high temperatures during the first 6-12 months of use. Potential impurities are boron, silica, sodium, and traces of lead, arsenic and potassium. Not surprisingly, hard glass is relatively expensive, difficult to install, and subject to breakage. Glass-lined storage tanks are not common and the lining is subject to failure over an extended period of time.

Fuzed quartz is sometimes used for high purity water distribution in local areas and for high purity water still condensers. It is superior to hard borosilicate glass. Quartz is heat resistant and rivals block tin as a desirable inert material in contact with pure water. Quartz is very expensive, hard to work with, and has limited practical uses.

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Stainless Steel. Stainless steel is a practical material used in high purity water distribution and storage facilities. Type 304 or 316 low carbon stainless steel is normally specified. Stainless steel has high strength and is relatively resistant to attack by pure water. Stainless steel tanks are popular and are relatively low in cost. Iron, chromium, and nickle are the main stainless steel impurities which normally leach into solution. Initial flushing of the stainless steel storage tank or distribution system with an acid solution is normally recommended to remove oxide scales or impurities on the outside of the pipe. Stainless steel is very hard, making pipes and fittings difficult to install.

Aluminum. Next to block tin, aluminum is one of the purest metals commonly used to convey or store high purity water. Type 1060 which is normally specified contains only 0.4 percent impurities which are mostly copper, silver, and magnesium. From a biological standpoint it is a relatively non-toxic metal. Unfortunately, it is active electrolytically. If in contact with other metals such as stainless steel, aluminum will corrode and go into solution along with its impurities. The interior surface of aluminum piping is fairly porous and may harbor biological activity (similar to PVC pipe). Aluminum does have the advantage of being able to withstand high temperature so the user can sterilize the system with pure steam.

Distribution

When referring to a distribution system, some sort of pipe network is normally visualized. In practice, this is not always the case. Many hospitals store and dispense water from containers carried throughout the hospital which are filled at a central supply. A pipe distribution network is preferred over this type of distribution. Disadvantages of a "container network" are that it incorporates a middle man (to fill and transport the containers), often offers stale water to the user, and introduces the potential of contamination of the water from microorganisms in the air of the hospital.

Earlier it was noted that a desirable goal of any distribution system is to get freshly produced water quickly to the user. This implies that large, complicated high purity water networks are not desirable. There is more time for the high purity water to sit in long lengths of pipe and become contaminated from leaching of pipe materials, growth of microorganisms, or carbon dioxide and other contaminants in the air. The quality of water within the distribution system will vary considerably. There are many dead ends.

Ultimate size of the pipe network is a function of production costs and use requirements. Whatever the size, continuous large-loop recirculation reduces degradation and dead ends. It allows for continuous monitoring of the freshly produced water (with on-line analysers such as a conductivity meter) and quick cross-connection detection.

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For recirculation to be effective it must involve the entire distribution system. That is, water in the entire network must be continuously replaced and retreated. Flow does not have to be large, just enough to avoid stagnation, about three times the volume in the network per hour. Furthermore, the flow won't exert a significant load on ion exchange filters or carbon beds because continuously recirculated waters pick up few contaminants. Recirculation is very important if the goal of providing a uniform quality high purity water to the user is to be attained.

Flow from the production site to the user is preferably by gravity. This avoids potential recontamination by the pump and is simpler and more reliable. It also implies that production and storage of high purity water should be on the top floor for a centralized system. If a booster pump must be used, the pump seals should be made of Teflon or equivalent and the impeller and casing of stainless steel or some inert plastic material. Centrifugal pumps are preferred since use (flow) varies and it is desirable to maintain constant pressure.

Distribution pipe should be sized according to peak (or at least "adequate") flow. When use of high purity water is substantial, having it come out of the faucet at low pressure will discourage indiscriminate use. Whatever the flow, it can be expected to be spasmodic because of the intermittent nature of high purity water use. Recirculation will somewhat dampen flow highs and lows.

Storage

In the case of high purity water, storage is not a desirable condition, it is a practical one. It is not always feasible to produce adequate high purity water quantities on demand where needed. The alternative is to store previously produced water.

Storage offers the most opportunity for water contamination because it affords the most time for contamination to occur. Basically, stored water must be protected from impurities leached from the storage tank, dissolved from the atmosphere, or (in the case of biological impurities) produced in the water itself. There is also the potential of a poor quality plug of water entering from the treatment system and contaminating the water in the tank.

The previous section on materials discussed the merits of common materials used in high purity water production. Storage tank materials include consideration of gasketing and pipe joining materials, coupling threads, and draw-off faucets also.

Air and particulate filters remove unwanted gas vapors, small particulates, and airborne bacteria from the outside air which replaces drawn-off

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water in the storage tank. The filters may contain filter material, activated carbon, and/or ion exchange resins to remove ionizable gases such as carbon dioxide. Air passes through the filters ("vent guards") only on demand. Care must be taken to size the air filters correctly or risk tank collapse during a heavy draw-off or draining. For special conditions, pure oxygen, argon, or nitrogen may be desirable to use as a gas blanket in lieu of purified air.

Ultraviolet lights are frequently placed in storage tanks. Ultraviolet light waves emitted between 230 and 300 nanometers exhibit germicidal effects. Most ultraviolet lamps emit a great portion of their ultraviolet light intensity at 254 nanometers. Ultraviolet lights are sheathed in quartz tubes because ultraviolet light will not pass through glass. Sheathing is necessary to enable replacement of the light without disturbing the contents of the tank.

The intensity of ultraviolet light diminishes quickly as it travels in water. The ultraviolet intensity will approach zero a few inches from the lamp. Furthermore, dissolved organic and particulate matter absorb ultraviolet light so that the stored water must be very clean. At best, ultraviolet lamps placed in storage tanks keep water sterile that is already sterile. If significant biological impurities are introduced into the water via the treatment process (for instance a contaminated ion exchange column) or the air, biological growth may occur even with ultraviolet light present. Recirculation of water through the storage tank is desirable to maintain a constant flow of fresh water passing through the effective ultraviolet intensity field. A more effective (and more costly) method to prevent regrowth of bacteria in a storage tank is to store the water at 180°F. Another alternative is to recirculate the stored water through a thin-film ultraviolet water sterilizer specifically designed for that purpose.

The average to peak flow ratio influences storage capacity requirements. The closer the ratio is to one, the less storage capacity is needed. The smaller it is, the more capacity is required. An alternative to large storage capacity is schedule restriction of flow rates. The practicality and desirability of flow scheduling is open to discussion.

Storage requirements are also affected by the type of high purity water treatment. Storage is less important for ion exchange or reverse osmosis systems where relatively large amounts of clean water can be recirculated at little increase in treatment costs. In distillation systems, treatment cost is nearly directly proportional to the quantity of water evaporated in the distiller, whether it be high purity recirculated water or incoming tap water.

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CENTRALIZED AND DECENTRALIZED PRODUCTION SYSTEMS

From an overall control and maintenance viewpoint, centralized treatment operations are more desirable than decentralized operations since there is less treatment equipment. On the other hand, centralized treatment operations are usually associated with large distribution networks. Such networks are more susceptible to water contamination from leaching, cross connections, and/or failure of the integrity of the network. In addition, if any portion of the centralized system (treatment or distribution) fails, all portions suffer. A centralized system also implies a single quality level.

Decentralized treatment operations allow precise control of the individual unit and, of course, have smaller distribution networks (if any). The extent of decentralization may influence who controls the high purity water production unit (the user or supplier). Operation, maintenance, and control activities (such as monitoring and surveillance of the high purity water) need to be agreed upon by all concerned parties. Decentralized treatment systems have high initial capital costs and (because of their numbers and size) incur high operating and maintenance costs also.

Other factors influence centralized/decentralized considerations. The implication of fairly uniform demand (average to peak flow ratios close to one) is conducive to centralized production. If average to peak flow ratio is considerably less than one, centralized production systems will probably require significant storage facilities. Irratic flow may foster stagnant areas in the distribution network and resulting high purity water quality will be less predictable. For such cases it is beneficial to adopt a local, decentralized production system.

Water quality requirements also affect production system selection. When water quality requirements are fairly constant throughout the user network, centralized systems are desirable. When there are varied quality requirements or critical end use points within the network, localized treatment is desirable.

A compromise approach is to utilize a semi-centralized high purity water production system. A semi-centralized system involves a functional breakdown of high purity water requirements on the basis of discipline and/or department. Each discipline and/or department is provided with its own system. An advantage of this approach is that in the event of a system break down, high purity water will be available from another system. A variation is one where the first line water treatment such as water softening or rough deionization is on a centralized system with the semi-centralized systems further purifying the water as needed.

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HIGH PURITY WATER USES IN MEDICAL FACILITIES AND LABORATORIES

High purity water use is affected by a wide variety of equipment, analytical instruments, and personal ideas of the medical and technical staffs. It is affected by the type of hospital (general, research, or teaching) as well as the hospital size. It is affected if water for intravenous solutions is produced at the hospital or purchased. Water for intravenous solutions must be of the highest purity. When produced on site, adequate precautions must be taken to insure uniform treatment and proper surveillance of water quality.

In general, high purity water is used for cleaning, as a diluent or solvent for reagent and culture preparation, and for direct or indirect patient use. Cleaning includes utensil washers of all types, sonic washers, and sterilizers. Operation of such equipment is normally affected by internal deposition caused by water hardness or corrosion caused by water acidity and other dissolved gases when the water is heated. It is important to understand alkalinity, acidity, and hardness relationships. Table 11 shows their basic associations. Table 14 gives general water softening criteria as well as Army requirements.

Equipment manufacturers instructions do not normally specify influent water criteria except to require drinking water quality or higher. Manufacturers commonly recommend detergents or cleansing agents to overcome "normal" water hardness and clean utensils properly. Less than 500 mg/l total solids is frequently considered "normal" amounts of solids encountered in water supplies (much of which is dissolved, hardness producing solids). A review of Table 14 implies that hardness of less than 50 mg/l as $CaCO_3$ is desirable for influent water to equipment generating hot water.

In addition to equipment requirements, attention should also be paid to final rinse water quality which may be a function of the intended use of the washed utensils. For instance, pyrogen free water is often used to give surgical instruments a final rinse.

Water purity affects chemical analyses and microbiological cultures. It may result in erroneous interpretation or identification of results. The frequently used high purity water standards are adequate for most common analytical uses. Additional special requirements must be identified by the investigator and proper measures taken to reduce the concentration and/or regularly monitor the offending impurities. Biomedical testing and research is an area where special precautions must be taken. Table 16 lists some common medical laboratory activities and contaminants which (in sufficient quantities) may affect their outcome.

The most obvious direct uses of high purity water are for irrigation or injection. USP standards address water quality requirements for such uses in detail and they are summarized in a previous section. Indirect

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TABLE 16. WATER IMPURITIES INTERFERING WITH MEDICAL LABORATORY ANALYSES

Contaminant	Potential Activities Affected
Metals	Enzyme activity Tissue cell culture Biological growth and analyses Chemical analyses Nutrient preparation Glassware cleaning Conductivity measurements Paper chromatography separation Histological slide preparation
Organics	Enzyme activity Biological growth and analyses Hemoglobin analyses Glassware cleaning Conductivity measurements
Particulates	Microscopic identification Colorimetric determinations
Dissolved gases	Biological growth and analyses Small volumetric measurements
Salts	Enzyme activity Biological growth and analyses Conductivity measurements Amino-acid analyses Atomic absorption measurements Stain preparation
Microorganisms	Enzyme activity Tissue cell culture Nutrient preparation Conductivity measurements Pyrogen production Trace metal analyses Reagent stability Stain interpretation

uses of high purity water include use in inhalation therapy devices and in dialysis fluids. The association for the Advancement of Medical Instrumentation (AAMI) has published a standard which establishes requirements for materials, components, monitors, accessories, maintenance and labeling of artificial kidneys. Water purification requirements recommended by AAMI are summarized in Table 17. Indirect uses also include high purity water for bulk internal liquids (like cough medicines) or to supplement oral antibiotics. Table 18 lists typical health care activities or equipment requiring high purity water considerations.

Quantity of high purity water will vary with the hospital size and services offered. Consumption is normally expressed in gallons per bed. In general, the more services provided (especially significant laundry services or extensive laboratory and research activities), the greater will be the total use of high purity water. In addition, the larger the hospital bed capacity, the lower will be the water required per bed. One estimate of high purity water use is 0.1 gph per bed. This compares with an average 10 gph per bed of tap water use.

For any particular situation a health facilities planner or hospital consultant must be employed when estimating water use requirements since they will vary greatly. Daily versus peak high purity water requirements affects water production equipment sizing and storage capacity. Additional flows due to recirculation and wasting (distiller blow down, reverse osmosis brine bleed-off) must be taken into account. Production of water for use in parenteral solutions requires the very highest purity and the most stringent monitoring activity.

CONTROL OF HIGH PURITY WATER QUALITY

The production of high purity water serves no useful purpose if there is lack of confidence in its quality and alternate sources are used. Confidence in product water quality is gained only when representative samples are tested at regular intervals, when the results of the tests are distributed to interested parties, and when quick action is taken to remedy the problem if product water falls below specified limits.

Monitoring high purity water has several objectives. Surveillance of produced high purity water quality to insure its suitability for use is the most obvious. Source water quality should also be monitored periodically since changes in it will influence the efficiency and effectiveness of the high purity water production system. When source water is treated tap water, data kept by the local water treatment plant is a convenient way to supplement source water quality measurements. Detection of contaminants leached from or developed during production, storage, and distribution is a third objective. Providing criteria for proper operation and maintenance of the high purity water production operation is a fourth. These objectives are not mutually exclusive. They do provide some insight about desirable sampling locations and frequencies.

TABLE 17. SUMMARY OF WATER PURIFICATION REQUIREMENTS FOR HEMODIALYSIS
AS PROPOSED BY THE ASSOCIATION FOR THE ADVANCEMENT
OF MEDICAL INSTRUMENTATION

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1. Specific recommendations for water treatment in any locality should be made by the physician in charge.

2. Water treatment may be by deionization and/or reverse osmosis with influent water quality to meet US Public Health Service Drinking Water Standards.

    a. Deionization alone - effluent continuously monitored
                                  - effluent resistivity $\geq$ 1 megohm-cm

    b. Reverse osmosis alone - effluent continuously monitored
                                    - effluent resistivity $\geq$ 10,000 ohm-cm

    c. Combined treatment - meet requirements of a. above

3. Maximum chemical concentrations for purified water.

Substance	Maximum Concentration (mg/l)
Calcium	10.0
Magnesium	3.0
Potassium	8.0
Sodium	150.0
Fluoride	0.2
Chlorine	0.5
Chloramine	0.0
Nitrate	2.0
Sulfate	100.0
Trace metals (copper, mercury, barium, tin, zink, each)	0.1

4. Maximum microbiological concentrations for purified water.

    a. Standard plate count $\geq$ 100 colonies/ml.

    b. Samples taken at entrance to proportioner or mixing tank of artificial kidney machine.

    c. Samples assayed within 30 minutes of collection or stored at 5°C and assayed within 24 hours.

    d. Frequency of sampling:

        (1) At least once monthly for monitoring purposes.

        (2) Repeat samples taken when bacteriologic counts exceed the allowable level.

        (3) Collect sample when clinical indications suggest pyrogenic or septicemic complications.

    e. Standard plate counts should be determined using conventional laboratory procedures. Culture media used can be tryptic soy agar, Standard Methods agar, or blood agar. Colonies shall be counted after 48 hours of incubation at 37°C.

Standard: Hemodialyses Standard (Proposed), Association for the
                  Advancement of Medical Instrumentation, Arlington, VA (1976).

TABLE 18. HEALTH CARE ACTIVITIES OR EQUIPMENT
REQUIRING HIGH PURITY WATER

Major Hospital Area	Principal Activities or Equipment	Potential Water Quality Parameters of Concern
Nursing unit	Inhalation therapy devices Sterilizers Use of irrigation solutions Use of injectable solutions Liquid diet preparation	Hardness Sterility Pyrogens Organic/inorganic purity
Operating room	Scrubbing Use of irrigation solutions Use of injectable solutions Sterilizers	Sterility Pyrogens Organic/inorganic purity Hardness
Clinical laboratory	Chemical and physical measurement Microbiological assaying sterilizers/washers Analytical equipment requirements Sonic cleaning	Many and various; depends on test
Central materiel supply	Glassware washing and rinsing Syringes, surgical instruments washing and rinsing Sonic cleaning Supplying high purity water to other hospital areas Sterilizers	Hardness Sterility Pyrogens Organic/inorganic purity
Kitchen	Pot/pan washers, glassware washers, dishwashers	Hardness
Pharmacy	Preparing water for injection, irrigation, bulk internal liquids, and oral antibiotics Sterilizers Washers	Sterility Pyrogens Organic/inorganic purity Particulates

A monitoring program must provide several items of information. It must specify where samples are to be taken and how frequently. It must define which tests are to be performed and by what method. Accurate records of water quality measurements must be kept and results reported. The results should be supplemented with information about the rate of high purity water production, dates of maintenance, servicing, and changes. Graphs or charts of all measurements should be plotted to record changes as well as absolute values. The College of American Pathologist Water Specifications outline a detailed water testing program. Items which they discuss should be considered for incorporation in any high purity water monitoring schedule.

Sampling at the users tap is the most realistic sampling location. Where there are many taps, random selection of tap locations at regular sampling intervals is a desirable approach. Less frequent sampling should also be done at the treatment train effluent to provide adequate information as to whether potential contaminants are leaching from the storage/distribution system or are coming from the high purity water treatment operation. Finally, sampling source influent water on a regular basis (and/or obtaining suitable data from the water treatment plant) is desirable since changes in source water will affect treatment operations as indicated previously. Data to insure proper day to day operation of the high purity water treatment equipment can be obtained either from the users tap where there is recirculation (and before storage), or at the treatment train effluent.

On-line water quality monitors are very desirable and are most appropriate in the continuous recirculation loop. The most common of these is measurement of the waters electrolytic content as resistivity or its reciprocal, conductivity. High purity water pH and turbidity are other common on-line water quality meters. Probes which measure oxidation/reduction potential and various anions and cations in solution are also available. The advantage of on-line water quality monitors is that they are automatic, provide a continuous measurement of water quality, and signal changes from the norm. Disadvantages are that they measure only at a set point, may be difficult to calibrate (and to stay calibrated), and that their ruggedness and reliability may be questionable. For very high purity water, specific contaminant levels may be below probe detectable limits.

Initial discrete sampling will be more frequent until the desired degree of confidence is established in the high purity water treatment system. After this initial period, frequency of sampling will become more a function of the water use. Identification of the water use implies that the high purity water criteria is specified. Critical high purity water constituents require most frequent monitoring. On the average, water should be sampled at least once per week in a continuous operation and the most critical impurities measured. Samples should be taken at least once monthly for other impurities. Samples should be taken after

cleaning or servicing any water treatment apparatus. For a batch operation, samples should be taken for each batch of water processed. When appropriate, discrete grab samples should be supplemented with on-line continuous "gross quality" analyses (such as conductivity or pH) which will quickly signal general water quality changes.

Water contaminants which most affect the water use must be measured directly. On these hinge the success or failure of the high purity production system. Obviously, if they are above tolerable limits or there is no confidence in their absence, the produced high purity water will not be used or be subjected to further treatment. Additional treatment is, of course, perfectly acceptable and most likely to be used when several high purity water qualities or types are needed through a facility. It is when such additional treatment is unscheduled, redundant, or unnecessary (according to design) that the objectives of the high purity water system are not being met. Such cases emphasize the need for confidence to be established in the high purity water system by constant monitoring and reporting of results.

Specific contaminants which need to be monitored depend on the use of the water or the test to be run. Thus, they are identified by the user/investigator or some high purity water criteria. Specific contaminants which actually are measured include other considerations in addition to water use. These are ease of measurement of a contaminant, available equipment or laboratory to measure the contaminant, available expertise, time, and money. These practical considerations must be faced if a viable monitoring program is to be maintained which is acceptable to all parties.

Common high purity water criteria outline which contaminants should be measured so that the high purity water defined will meet most user requirements. They also detail measurement procedures. To varying extents, additional information with respect to frequency of testing, sample gathering, handling, and storage is given. If one of these high purity water criteria are adopted, all applicable guidelines should be followed. The most common criteria have been outlined and referenced in a previous section on this subject.

Source water impurities which affect high purity water treatment facilities (hardness, alkalinity, etc.,) have been identified and discussed in the section on equipment operation. "Standard Methods for the Examination of Water and Wastewater" or the Environmental Protection Agency's "Manual of Methods for Chemical Analysis of Water and Wastes" provide additional guidance for measurement of impurities other than by on-line equipment. In addition to outlining measurement procedures, these manuals also specify and discuss the apparatus required for measuring the water quality such as carbon analyzers, spectrophotometers, and colorimeters.

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